

Sampling, Analysis, and Quality Assurance Plan

Former Earth Protection Services, Inc.
Phoenix, Arizona

Prepared for:
Mr. John Chilcott, President
VJ2C, Inc.
Phoenix, Arizona

April 2011

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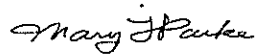
EPSI

Sampling, Analysis, and Quality Assurance Plan

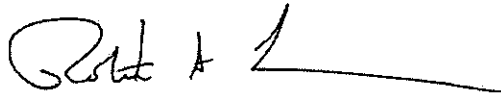
Former Earth Protection Services, Inc.
Phoenix, Arizona

April 2011

Project No. 111988



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**ATTACHMENT A - COPY OF USEPA CONDITIONAL APPROVAL FOR FORMER
EARTH PROTECTION SERVICES INC. FACILITY, SUITE 4,
10 SOUTH 48TH AVENUE, PHOENIX ARIZONA, TSCA
PCB CLEANUP UNDER 40 CFR 761.61 (A) AND 761.61 (C),
DATED MARCH 18, 2011**

ATTACHMENT B - ACCUTEST QA MANUAL

**ATTACHMENT C - USEPA COMPENDIUM METHOD TO-10A, COMPENDIUM OF
METHODS FOR THE DETERMINATION OF TOXIC ORGANIC
COMPOUNDS IN AMBIENT AIR, SECOND EDITION,
USEPA/625/R-96/010B, JANUARY 1999.**

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LIST OF ACRONYMS

µg/100cm ²	micrograms per 100 square centimeters
µg/L	micrograms per liter
ADHS	Arizona Department of Health Services
CAFO	Consent and Final Order
DQO	Data Quality Objectives
EPSI	Earth Protection Services Inc.
HSP	health and safety plan
LCS	Laboratory Control Sample
LCSD	Laboratory Control Standard Duplicate
LRL	Laboratory Reporting Limit
MS	Matrix Spike
MSD	Matrix Spike Duplicate
mg/kg	milligrams per kilogram
NELAC	National Environmental Laboratory Accreditation Conference
PCBs	polychlorinated biphenyls
PQL	Practical Quantitation Limit
RPD	relative percent difference
RCRA	Resource Conservation and Recovery Act
QA/QC	Quality Assurance/Quality Control
SIP	Self-Implementing Procedure

TSCA Toxic Substances Control Act

USEPA United States Environmental Protection Agency

This Sampling, Analysis and Quality Assurance Plan (Plan) has been prepared by ERM on behalf of Earth Protection Services, Inc. (EPSI). Until recently, EPSI operated an electronics recycling facility at 10 South 48th Avenue, Suite 4, in Phoenix, Arizona (Site). During its operations, EPSI accepted polychlorinated biphenyl (PCB) and non-PCB containing fluorescent light ballasts for storage, processing, and recycling; consequently, EPSI operated at this location under a Toxic Substances and Control Act (TSCA) approval issued by the United States Environmental Protection Agency (USEPA). The approval was dated March 7, 2003, and was titled *TSCA Approval for PCB Commercial Storage Facility* (USEPA ID Number AZR 000 005 454). In accordance with 40 CFR 761.61 (a), a Self Implementing Work Plan (SIP), titled Revised Work Plan Draft Former Earth Protection Services Inc. (EPSI) Facility Phoenix Arizona, dated February 7, 2011 was submitted to Region IX USEPA, and the USEPA Conditional Approval for Former Earth Protection Services Inc. Facility, Suite 4, 10 South 48th Avenue, Phoenix Arizona, TSCA PCB Cleanup Under 40 CFR 761.61 (a) and 761.61 (c), dated March 18, 2011, was received by EPSI on March 22, 2011 (Conditional Approval).

EPSI is currently seeking closure of the facility in accordance with 40 CFR 761.61 (a) and 761.61 (c). EPSI has already completed site assessment, demolition, decontamination, and sampling activities as summarized in Table 1. This Plan is intended to serve as a guide for additional sampling and laboratory testing of porous and non-porous surfaces, as well as soil and sediment, during the closure of the EPSI facility. The primary objective of this sampling is to provide a description of the additional characterization sampling of porous and non-porous surfaces, soil, and PCB-containing dust. If the additional characterization sampling data shows levels in excess of the 1 milligram per kilogram (mg/kg) (porous surfaces) or 100 ug/100 sq. cm (non-porous surfaces), then additional cleanup will be required. In accordance with the Conditional Approval, EPSI will achieve the cleanup level for concrete equal to or less than 1 mg/kg PCBs in the upper 0.1 to 1.0 inch layer of the concrete floor and certain walls inside Suite 4. Indoor and outdoor air sampling will be conducted following completion of PCB characterization and remediation to assure compliance with the USEPA risk based air standards referenced in the Conditional Approval.

Table 1 ***Timeline of EPSI Site Characterization and Assessment Activities***

Date	Activity
February 2009	EPSI informs USEPA Region IX of intent to close facility.
February 18, 2010	Wipe Sampling of former Processing Equipment (freezer, block wall, magnet, bins, etc.).
March 2010	All processing equipment is removed from site, except Freezer.
Week of July 26, 2010	Removal of concrete block walls that surrounded the Ballast Processing Room, freezer and electrical conduit and water sprinkler lines supplying the freezer, drywall on the north wall of the Ballast Processing Room, electric and compressed air piping/conduits supplying the Ballast Processing Room were removed back to the panel.
Week of August 1, 2010	Secondary containment curbing, ramps and removal of the floor coating (top ¼ inch removed including coating and concrete) in the Ballast Storage area (Area A1), only. Floor and remaining walls cleaned by power washing with water and an industrial cleaning solution.
August 10, 2010	Limited wipe and shallow concrete sampling of cleaned areas.
Week of August 30, 2010	Removal of wood expansion joint, saw cut 2-foot by 2-foot opening and remove top 3-inches of soil/fill material around electrical grounding rod.
September 15, 2010	Collected core samples immediately adjacent to the shallow concrete samples through the slab and re-sampling the soil around the grounding rod.

Until recently, EPSI operated an electronics recycling facility at 10 South 48th Avenue, Suite 4, in Phoenix, Arizona. A vicinity map showing the general location of the Site is included as Figure 1, and a Site Plan is included as Figure 2. EPSI first began operations at the Site in 1997. During its operations, EPSI accepted PCB and non-PCB containing fluorescent light ballasts for storage, processing, and recycling. Because the EPSI facility accepted PCB-containing materials, the facility operated under a TSCA approval issued by USEPA. The approval was dated March 7, 2003, and was titled TSCA Approval for PCB Commercial Storage Facility (USEPA ID Number AZR 000 005 454).

The former EPSI facility is located on approximately 1.3 acres of land and includes 21,500 square feet of office/warehouse space, as well as ancillary paved parking and drive areas, landscaped areas, and unpaved areas. The office/warehouse space can be divided into eight separate areas (seven process areas and the office area) based on the homogeneity of the former activities conducted within the facility, as follows:

- A1 Ballast Storage Area
- A2 Freezer Area
- A3 Ballast Processing Area
- A4 Material Staging Area
- A5 Lamp Area
- A6 Walkway Area
- A7 Miscellaneous Storage Area
- A8 Office Area

During EPSI's operations, light ballasts were received, staged, stored, and reprocessed/recycled at the facility. The general process followed by EPSI was as follows:

Containers were received by EPSI from generators. Containers were separated depending on container labels. Containers labeled by generator as not containing ballasts with PCBs were stored in an area separate from containers labeled as containing ballasts with PCBs.

- As needed, ballasts were transferred from non-metal drum containers into metal drums.

- Drums were moved into a freezer and kept in the freezer for approximately 8 hours to allow the potting compound inside the ballasts to solidify.
- After the potting compound solidified, the drums were moved from the freezer into a ballast processing area and opened.
- Ballasts were then unpacked from each drum, and protruding wires were removed for recycling. The casing was then opened and the coil and capacitor removed. Capacitors and potting compound were placed in a metal drum to be shipped off the Site for disposal. Ballast cases and copper and aluminum coils were placed in separate bins to be shipped off the Site for recycling.

Potting compound and small capacitors removed from unlabeled ballasts and ballasts labeled as containing PCBs were either incinerated at a TSCA permitted facility or land filled at a RCRA permitted facility. No disposal or recycling of waste occurred at the Site. Wastes were transferred from the Site for disposal or reuse.

Removal and decontamination activities have already occurred at the Site as summarized in the *Revised Work Plan Draft Former Earth Protection Services Inc. (EPSI) Facility Phoenix, Arizona*, dated February 7, 2011. Additional sampling and site characterization is required by the Conditional Approval and described in the following sections.

3.0

SAMPLING AND ANALYSES PLAN (SAP)

3.1

PURPOSE

EPSI is currently seeking closure of its TSCA-permitted facility located at 10 South 48th Avenue, Suite 4, in Phoenix, Arizona in accordance with USEPA requirements. EPSI previously accepted PCB and non-PCB containing fluorescent light ballasts for storage, processing, and recycling at this facility. As part of the facility closure, EPSI is required to demonstrate the facility has been adequately decontaminated. This plan will serve as a guide for sampling and laboratory testing of porous and non-porous surfaces, as well as soil and sediment, during the closure of the facility. The sampling and testing is intended to confirm the facility has been sufficiently decontaminated and/or remediated.

PCB analytical results will be compared directly to the standards of 1 mg/kg and 10 micrograms per 100 square centimeters (ug/100 cm²) for total PCB Aroclors. The goal of this Plan is characterize potential PCB-impacted porous and non-porous surfaces, soil, and sediment exceeding the standard of 1 mg/kg (porous surfaces, soils, and sediment) or 10 ug/100 cm² (for non-porous surface) for total PCB Aroclors.

The project approach will consist of assessing those areas of the Site considered to have potentially been impacted by PCBs during the historic recycling activities conducted at the Site. These areas include the former process/material storage areas within the building interior; the valley gutter on the south and east sides of the Site where surface water is channeled; and the unpaved area on the west side of the Site that includes the two Consent and Final Order (CAFO) areas.

If PCB concentrations are identified at a given location exceeding the standard of 1 mg/kg or 10 ug/100 cm² for total PCB Aroclors, the location will be remediated/decontaminated as required in the Conditional Approval. Following completion of decontamination and/or remediation activities, (if required), additional confirmation/closure sampling and testing of the affected media will be conducted to confirm the effectiveness of the decontamination and/or remediation effort. Following the completion of the PCB characterization/remediation, air sampling will be conducted inside and outside Suite 4 to assure that the risk based indoor air standard for PCB Aroclors 1242 (0.021 to 2.1 microgram/cubic meter) is not exceeded.

Prior to beginning the field work, ERM will prepare a site-specific health and safety plan (HSP) for the anticipated Site activities. The plan will address worker safety issues, delineation of work zones, personal protective equipment (PPE), and other pertinent aspects of the work. The basic level of PPE to be used during the closure activities is OSHA Level D for outdoor activities and OSHA Level C for indoor activities. The additional characterization sampling proposed in the plan is summarized in Table 2.

Table 2 *Summary of Additional Characterization Sampling*

Location	Media	Collection Method	No. of Samples
Interior Building, Areas A1-A8	Concrete	Rotary impact hammer drill equipped with a 1-inch diameter carbide drill bit into the upper 0.5-inch o	181
Outdoors directly under Exterior Bay doors on South and East Sides of building	Asphalt	Rotary impact hammer drill equipped with a 1-inch diameter carbide drill bit into the upper 0.5-inch o	12
Exterior, North and South CAFO	Soil	Split-spoon sampler approximately 6 inches into the underlying soil.	6
Valley Gutter corresponding to the Exterior Bay doors on the South and East Sides of building	Concrete	Rotary impact hammer drill equipped with a 1-inch diameter carbide drill bit into the upper 0.5-inch o	6

3.2.1

Interior Surface Areas

Concrete samples will be collected at the frequency of 1 sample per 100 square feet, per USEPA's July 22, 2008 *Standard Operating Procedure for Sampling Porous Surfaces for Polychlorinated Biphenyls (PCBs)* for the floors in Areas A1-A7 floors and the wall in Area A3, as shown in Figures 3 and 4. In addition, one concrete sample will be taken on the inside of each entry door from the processing area in A8 (Figure 3). The concrete samples will be obtained using a rotary impact hammer drill equipped with a 1-inch diameter carbide drill bit. At each sample location, a sufficient number of holes will be drilled into the upper 0.5-inch of the concrete surface to generate a minimum of 40 grams of fine powder. As the holes are drilled, the concrete powder will be collected and placed into a laboratory-prepared 4-ounce glass sample jar with a Teflon-lined cap.

Based on the results of the concrete chip samples, if PCBs are measured in excess of the 1 mg/kg, EPSI will collect soil samples from below 1-inch diameter cores by drilling through concrete slab with a coring machine. A minimum of 40 grams of soil will be collected below the concrete and placed into a laboratory-prepared 4-ounce glass sample jar with a Teflon-lined cap.

Once sampling of the porous surface is complete, the sample location will be patched using a quick setting concrete. The owner of the Site building will be made aware of the planned sampling activities and the method of repairing the floors and walls at least seven days prior to starting sampling activities.

For the areas inside the Site building (Areas A1 through A7), if all samples from a surface in an area contain total PCB Aroclor concentrations that are less than 1 mg/kg (concrete) or 10 ug/100 cm² (wipe sample), that surface will be considered clean. These samples will then be considered closure samples and will be submitted for data verification/data validation.

If one or more samples from a surface in an area inside the building exceed the cleanup standards of 1 mg/kg (for concrete samples) or 10 ug/100 cm² (for wipe samples) total PCB Aroclors, this surface will be decontaminated and the surface re-sampled by collecting samples adjacent to the original sample locations for this surface. Once all samples from a surface in an area inside the Site building contains a total PCB Aroclor concentration less than 1 mg/kg (concrete, soil and asphalt) or 10 ug/100 cm² (wipe samples), the samples will be considered clearance samples.

3.2.2 *Soil Samples – West Side and Former North and South CAFO*

To characterize the west side of the building, three samples will be collected along the width of each exterior bay doors on the west side of the building (coinciding with the north and south CAFO) in the area designated as A9, as shown in Figure 4. Proposed sample locations will be initially biased towards areas of oily or stained debris. At each sample location, surficial debris/material will first be cleared away from the proposed sample location, and the split-spoon sampler will then be driven approximately 6 inches into the underlying soil. Once extracted from the borehole, a representative portion of the soil sample will be removed from the split-spoon sampler and placed into a laboratory-prepared 4-ounce glass sample jar with a Teflon-lined cap.

3.2.3 *Asphalt Samples – Exterior Bay Doors*

Asphalt samples will be obtained on the exterior of the building, adjacent to the exterior bay doors, as shown in Figure 5. Samples will be collected from the asphalt surfaces directly below the doors in areas that appear stained, cracked or damaged. If no stains, cracks, or damage are evident, three evenly spaced samples will be collected along the width of the door. A rotary impact hammer drill equipped with a 1-inch diameter drill bit will be used to core to a depth of 0.5-inch of the concrete surface to generate a minimum of 40 grams of material. The sample will be placed into a laboratory-prepared 4-ounce glass sample jar with a Teflon-lined cap.

Based on the results of the asphalt chip samples, if PCBs are measured in excess of the 1 mg/kg, EPSI will core a 2-inch diameter cores by drilling through asphalt with a coring machine. A minimum of 40 grams of soil will be collected below the asphalt and placed into a laboratory-prepared 4-ounce glass sample jar with a Teflon-lined cap.

3.2.4 *Concrete Samples – Exterior Bay Doors*

Six samples will be obtained locations along the valley gutter (shown as Area A10, Figure 5) corresponding to the asphalt samples taken in Section 3.2.3 of the Exterior Bay Doors, or in areas that appear stained, cracked, or damaged. EPSI will core to the bottom of the joint that appears to be damaged or cracked and collect a sample from the bottom of the damaged joint or crack. If the damaged joint or crack is found to extend the entire thickness of the concrete, EPSI will collect a sample of the underlying soils.

3.3 *PRE-CLEANUP WIPE AND DUST SAMPLES*

Prior to the commencement of any cleanup activities, the internal roof over the top of the office area (A8) will have wipe samples collected in at least three areas, as shown in Figure 3. Wipe samples will be collected in general accordance USEPA's *Wipe Sampling and Double Wash/Rinse Cleanup* as Recommended by The Environmental Protection Agency PCB Cleanup Policy, dated June 23, 1987 (revised and clarified on April 18, 1991). The location of each wipe sample will be framed using a 100 cm² template. The wipe sample will then be obtained by wiping the sample surface using a laboratory-prepared 2-inch square piece of sampling gauze saturated with hexane. Using uniform pressure, the sample surface will be wiped from left to right in rows from top to bottom, and again from top to bottom in

columns from left to right. Once the area has been wiped, the sampling gauze will be placed in a 4-ounce glass sample jar with a Teflon-lined cap.

In addition to the wipe sample collections, 1 bulk sample of dust will be collected from the Area A8 internal roof. A sample will be collected using a new whisk broom and a dust pan and placed into a laboratory-prepared 4-ounce glass sample jar with a Teflon-lined cap.

3.4 CONCRETE CLEANUP VERIFICATION SAMPLING

A table summarizing concrete verification samples to demonstrate after cleanup the PCBs in concrete do not exceed the USEPA approved cleanup level will be provided within 21 calendar days after implementing decontamination procedures in accordance with the Conditional Approval, Condition C (3)(c). EPSI proposes to use the same spacing, depth and utilized in the Additional Characterization Sampling plan (one sample per 3-meter square grid, 0.5 inches depth). The number of samples will depend on the area requiring cleanup.

3.4 SOIL AND ASPHALT CLEANUP VERIFICATION SAMPLING

A table summarizing soil and asphalt verification samples to demonstrate after cleanup the PCBs in concrete do not exceed the USEPA approved cleanup level will be provided within 21 calendar days after implementing decontamination procedures in accordance with the Conditional Approval, Condition C (3)(d).

3.5 POST CLEAN UP AIR SAMPLING

If cleanup is required, EPSI will collect all dust generated by cleanup activities with dry vacuum methods. The dust will be segregated, properly packaged, and profiled for disposal purposes. If the dust is in excess of 1 mg/kg PCBs, it will be disposed of in a Class I landfill. Depending on the extent of cleanup activities inside and outside Suite 4, post clean up air sampling will consist of a minimum of 3 sets of sampling in any area that dust was generated. USEPA Method TO-10A or TO-4A will be used for the collection and analyses of the indoor and outdoor samples. A copy of this method is attached in Attachment B

3.5

LABORATORY AND SAMPLING METHODOLOGY

The analytical laboratory for this project (Accutest) is a National Environmental Laboratory Accreditation Conference (NELAC) laboratory, and is also licensed by the Arizona Department of Health Services (ADHS) to perform the analyses required for this project. If, during the project, the status of the laboratory's NELAC or ADHS status changes, or if laboratory performance is not acceptable, an alternate laboratory with appropriate credentials will be selected to complete the testing. The laboratory is required to notify ERM if testing is to be subcontracted to another laboratory.

3.5.1

Laboratory Methodology

The analytical methods, sample containers, preservation, and holding times, and the cleanup levels for the various media to be sampled are summarized in the table below:

Table 3 Laboratory Analytical Methods

Extraction/ Analytical Method	Media	Container	Preservation	Minimum Sample Weight/ Volume	Extraction Holding Time/ Analysis Holding Time	Cleanup Levels
PCBs using USEPA Methods 3550C/8082A	Porous Surfaces (e.g., concrete) ea	4-ounce glass jar with Teflon- lined cap	Chill to 4°C	30 grams	14 days/40 days ²	< 1 mg/Kg
PCBs using USEPA Methods 3550C/8082A	Non- Porous Surfaces (wipe samples)	4-ounce glass jar with Teflon- lined cap	Chill to 4°C	Not Applicable	14 days/40 days ²	< 10 ug/ 100 cm ²
PCBs using USEPA Methods 3550C/8082A	Soil	4-ounce glass jar with Teflon- lined cap	Chill to 4°C	30 grams	14 days/40 days ²	< 1 mg/Kg
PCBs using USEPA Methods 3550C/8082A	Sediment	4-ounce glass jar with Teflon- lined cap	Chill to 4°C	30 grams	14 days/40 days ²	< 1 mg/Kg
PCBs using USEPA Methods 3550C/8082A	Asphalt	4-ounce glass jar with Teflon- lined cap	Chill to 4°C	30 grams	14 days/40 days ²	< 1 mg/Kg

Extraction/ Analytical Method	Media	Container	Preservation	Minimum Sample Weight/ Volume	Extraction Holding Time/ Analysis Holding Time	Cleanup Levels
PCBs using USEPA Method TO- 10A or TO-4A	Air	Poly- urethane Foam Filter (PUF)	Chill to 4°C	—	—	0.021 to 2.1 ug/cubic meter

Holding times for extraction and analysis begin the day of sample collection; therefore, the samples must be extracted or sample preparation must begin before the holding time is exceeded. Typically, the test is considered complete only after all analytical runs, including dilutions, quality control samples, and required re-analyses, are completed. For the proposed PCB test method (i.e., USEPA Method 8082A), the holding time is 14 days for extraction and 40 days for analysis.

A completed chain-of-custody form will be maintained from the time of sample collection until the samples arrive at the analytical laboratory. Samples will remain in the custody of the sampler until they are relinquished to another ERM employee or to the analytical laboratory. The chain-of-custody form will accompany the samples at all times, and will include the project number, name of sampler, sample identifications, sample matrices, dates and times of collection, number of containers, and analyses requested. The chain-of-custody form will be maintained by having the chemist/sample receiving clerk sign the chain-of-custody form upon receipt of the samples. The samples will be carefully packed in coolers with ice prior to transportation to the analytical laboratory.

The laboratory is expected to provide a ten business-day turn around for the results with an additional five working days to provide electronic deliverables.

3.5.2 *Sampling Methodology*

Sampling conducted at the Site will include porous (concrete and asphalt) and non-porous surfaces, as well as soil and sediment. The samples shall be obtained using clean sampling equipment which will be decontaminated by washing with laboratory grade detergent and water, rinsing with deionized water, and final rinsing with hexane. If a significant period of down-time is anticipated between successive sample collection, the sampling equipment will be placed in a plastic bag to reduce the exposure to potential airborne contaminants.

Regardless of the sample media (concrete, wipe, sediment, soil sample), immediately after obtaining each sample, a gummed custody seal will be placed across the jar and cap in a manner which would indicate tampering. Custody seals will be labeled with the sample identification number, the date and time of collection, the project number, and the initials of the person who obtained the sample. Once sealed and labeled, the samples shall be placed in a sample cooler with ice, and will be delivered at the end of each sampling day to Accutest for analysis. Each sample will be logged on a chain-of-custody form which will be kept with the samples until they are delivered to the laboratory. Recorded on the chain-of-custody form will be the name and signature of the sample collector, sample numbers, signatures of the persons involved in the chain-of-custody, the dates and times of custody and the tests to be performed. Once delivered to the laboratory, the chain-of-custody form shall be kept by the laboratory-dedicated quality assurance/quality control (QA/QC) representative.

4.0 QUALITY CONTROL PROCEDURES

4.1 MEASUREMENT OF DATA QUALITY OBJECTIVES

USEPA has established Data Quality Objectives (DQOs) to use as a guide for data quality assessment. The DQOs use data quality indicators for precision, accuracy, representativeness, completeness, and comparability to establish qualitative and quantitative objectives to ensure the data generated meet the needs of the project. According to USEPA's *Guidance on Systematic Planning using the Data Quality Objectives Process* (USEPA QA-G4), dated February 2006, the DQOs depend on the intended uses of the data and are based on the premise that the ultimate use(s) of a particular data set should dictate the quantity and quality of data. USEPA defines the data quality indicators as follows:

Precision is a measure of mutual agreement among individual measurements of the same property, usually under prescribed similar conditions expressed generally in terms of the standard deviation. Precision is often calculated by determining the relative percent difference (RPD) between duplicate samples. The proposed precision objective for sample duplicates is an RPD of 30 percent.

Duplicate Sample Conc. - Original Sample Conc.
Precision = $\frac{\text{Duplicate Sample Conc.} - \text{Original Sample Conc.}}{\text{Original Sample Conc.}} \times 100$

Accuracy represents another measure of data quality. It is the degree of agreement of a measured value of the analyte with its true or actual value. Accuracy includes a combination of random error (precision) and systematic error (bias) that are due to sampling and analytical operations. For analytical samples, accuracy is calculated as the percent recovery for a sample spiked with the analyte of concern (laboratory control samples [LCS], surrogates, or matrix spikes).

Amount of Analyte Recovered - Amount of Analyte Originally in Sample
Accuracy = $\frac{\text{Amount of Analyte Recovered} - \text{Amount of Analyte Originally in Sample}}{\text{Amount of Analyte Added to Sample}} \times 100$

Representativeness is a measure of the degree to which data accurately and precisely represents a characteristic of a population, a parameter variation at a sampling point, a process condition, or an environmental

condition. For this project, representativeness will be accomplished using consistent field sampling and analytical procedures.

Completeness is a measure of the amount of valid data obtained from a measurement system compared to the amount that was expected to be obtained under correct, normal conditions. For this project, the completeness objective will be 90 percent as determined using the following equation, and will relate to each surface sampled in an area.

$$\frac{\text{Number of Validated Measurements}}{\text{Total Number of Planned Measurements}} \times 100 = \text{Percent Completeness}$$

Comparability is a measure of the confidence that one data set or method can be compared to another. Since comparability cannot be measured quantitatively, we will use our professional judgment to evaluate this indicator. We will use consistent sampling and testing techniques and methods, so test results can be compared with a high degree of confidence.

The **Practical Quantitation Limit (PQL)** is the lowest concentration of a given analyte that can be reasonably achieved within the specified limits of precision and accuracy for routine laboratory tests. The PQL is also known as the laboratory reporting limit (LRL). All analytes detected at a concentration greater than or equal to the PQL will be reported.

The laboratory conducting the proposed analytical services is Accutest of Phoenix, Arizona. Additional information regarding qualifications and quality management is presented in Accutest's Quality Assurance Manual in Attachment A.

Documents will be stored at ERM's office for a period of two years after completion of the project. Older documents will be stored at a secure storage facility in accordance with ERM's records retention policy, for a minimum of nine years. The ERM Project Manager will be in charge of the storage of project-related documents. It is assumed documents submitted to USEPA will be retained by USEPA in accordance with its records retention requirements.

4.2

QUALITY CONTROL REQUIREMENTS

Additional QC samples can be obtained for use in evaluating the laboratory precision (sample duplicates), the effectiveness of equipment decontamination procedures (equipment blanks and field blanks), and

sample handling and transportation procedures (temperature blanks). The laboratory QC procedures are described in Accutest's Quality Assurance Manual in Attachment A. Other QC efforts not described in this plan may be implemented as part of the sampling and testing procedures used for the work.

In general, the following QC samples will be collected in the field.

Duplicate Samples: Field duplicate samples will be obtained at a frequency of 10 percent of the total number of samples obtained to assess laboratory precision. The duplicate samples will be obtained from a location adjacent to the original sample location, and they will be designated with a unique sample identification number, so the laboratory will be unaware that they are duplicates. At each sample location where a duplicate sample is to be collected, the quantity of matrix to be sampled will be doubled. The total quantity will be placed in a stainless steel bowl and mixed to produce a relatively homogenous matrix. The matrix will then be transferred into two sample containers.

Equipment and Field Blanks: Equipment and field blanks will be submitted to the laboratory for analysis. One of each type of blank will be submitted per sampling day as represented by one chain-of-custody document. The laboratory QC procedures will include additional analyses to assess the validity of the analytical results. These additional analyses will be conducted in accordance with *SW-846 7000A/8000B Quality Control Section* (USEPA 1992/2007). The additional analyses will include method blank analysis, laboratory control samples analysis, surrogate spike analysis, and matrix spike and matrix spike duplicate (MS/MSD) analysis (duplicate analysis). A description of these analyses is provided in the following paragraphs.

- method blank is a "clean," analyte-free sample (e.g., organic-free or deionized) water carried through the same entire sample preparation blank as an uncontaminated sample of the same matrix as the field samples (solid or liquid) which is analyzed in exactly the same, entire sample preparation and analysis procedure used for samples. The method blank measures the overall levels of contamination for the method. If the analysis indicates concentrations of target compounds in the method blank are greater than the practical quantitation limit (PQL) or the laboratory reporting limit, corrective action will need to be performed to identify the source of contamination.
- A MS sample is a field sample, which is spiked with a known concentration of a target analyte prior to sample preparation and analysis. It is used to determine the bias of a method for a particular

sample matrix. A matrix spike duplicate (MSD) is a second matrix spike sample spiked in the same manner as the MS. It is used both as a measure of accuracy and precision. For this project, the laboratory will be instructed to obtain the MSs and MSDs from the Site samples submitted for analysis.

- A LCS is a "clean," analyte-free matrix sample (e.g. organic-free or deionized water) spiked with a known concentration of the target analytes and carried through the same, entire sample preparation and analysis used for samples. The LCS is used to monitor the performance of a laboratory system. If the analysis indicates an LCS outside of the recovery acceptance limit, then corrective action will be required.
- A surrogate spike or analyte is a substance with properties that mimic or behave chemically similar to the analytes of interest. These compounds are added to every blank, sample, MS, MSD, and standard, and since they are not likely to be found in environmental samples, they are used to establish if the method has been performed properly.

Specific calibration and quality control criteria for the various test methods conducted by Accutest are identified in its Quality Assurance Manual in Attachment A.

4.3

DATA ACQUISITION REQUIREMENTS

The purpose of this plan is to assure the data obtained in the field and laboratory is suitable for the intended use; therefore, data will be evaluated at each step in which it is obtained, and the data will be compared to the quality criteria set forth herein. Field methods and procedures will be evaluated by the ERM employee(s) conducting the sampling. If problems are identified, they will be discussed with the Project Manager and/or the Project Principal. If data are found to be unusable, then additional samples may be obtained if required to characterize the extent of contaminants at the Site.

Data review, validation, and verification will be conducted by ERM and a Validation Subcontractor (LDC, Inc. or equivalent).

If laboratory control limits are exceeded, the data will be flagged or re-tested. If required by the Accutest Quality Assurance Manual, a corrective action report will be prepared and included with the data package provided to the data validation subcontractor. Data validation will further flag or qualify data based on their review. If the problems are significant,

corrective actions may include re-sampling and testing, or excluding the data in the final evaluation of the extent of contamination and remediation at the Site.

4.4 DATA MANAGEMENT

Following completion of the field activities, data review, and analysis will be conducted by ERM. The reviews shall be performed by qualified employees with understanding of the technical issues of the project. The Project Manager and Project Principal shall provide an additional final review for quality assurance purposes. Field data shall be reviewed to verify that laboratory data align with field conditions.

Data and information collected during the field work and generated by the laboratory will be checked and reviewed before being summarized in tables for the project reports. Field and analytical data will be reviewed by ERM staff for correctness, precision, accuracy, and completeness (as applicable). The Project Manager will check the data in the tables to verify that correct values have been entered. Data validation and verification will be conducted in accordance with the details presented in this plan.

4.5 DATA REVIEW, VALIDATION, AND VERIFICATION

Laboratory reports will be completed for each set of samples represented by a chain-of-custody form. These reports will be issued when the laboratory has completed the analyses and quality control review.

Initially all laboratory reports will include Level II documentation. The Level II data package will include the following:

- Name of client and laboratory.
- Cover letter with information on sample tests performed, a case narrative including problems encountered, and general comments.
- Analytical results reported by sample and by test with appropriate significant figures, and appropriate reporting limits that have been adjusted for dilution, if necessary. Also included will be appropriate information such as dates of analysis, date sampled, date extracted, sample matrix, analysis method, date received, and date reported.
- Identification of the test method(s) used.
- Quality control information including laboratory performance checks such as laboratory control spike, method blanks, MS, and MSD.

- A copy of the chain-of-custody form.
- Signature(s) of laboratory personnel responsible for analysis.

Once data is reviewed and laboratory results confirm the samples are representative of closure samples for a given area, a Level IV data package will be requested. The Level IV data package will include those items described below.

ERM will conduct a simplified Tier I data review on the laboratory data generated for this project. This review will include review of completeness of the data deliverables; the accuracy and completeness of the chain-of-custody; the condition of the samples upon laboratory receipt; the adherence to holding times and turn-around times; and issues with the analyses of the samples, project blanks, laboratory control samples, matrix spikes, reproducibility of duplicates, and surrogate recoveries.

For this project, 10 percent of the laboratory test results will also be evaluated using USEPA Level IV deliverables. A Level IV data package will be submitted by the laboratory to data validation subcontractor for a Tier IV review. The Level IV data package will include the information and data for a Level II data package and the following:

- initial and continuing calibration records;
- instrument and calibration blanks;
- system monitoring compound results;
- internal standard area and retention time summaries; and;
- raw data, including all instrumental printouts and worksheets/logs that contain information about how the test was conducted, including;
- daily tuning results;
- extraction, digestion, and sample preparation/cleanup logs;
- run/injection/sequence logs;
- chromatograms and instrumental quantitation reports; and
- intermediate calculation checks/worksheets/spreadsheets.

4.6

VALIDATION AND VERIFICATION METHODS

The Project Manager will review the daily reports, draft and final laboratory reports, and draft figures for consistency and compliance with this plan. Questions concerning the data will be answered at that time,

and the appropriate corrections will be made. Errors will be evaluated, and if necessary, additional samples will be obtained. ERM personnel will be required to follow the sampling program outlined in the Field Sampling Plan and the procedures described herein.

Verification of the laboratory reports by the Project Manager will be a cursory review of the report provided by the laboratory. Laboratory reports will be checked for the following:

- all of the samples were tested;
- the requested test method was used;
- sample holding times were met;
- a copy of the chain-of-custody form is attached and completely filled out;
- the laboratory reporting limits are within acceptable levels;
- laboratory quality control sample results are within the control limits;
- data flags or qualifiers are present; and
- the report is complete.

ERM staff will transcribe the laboratory data to tables and figures, along with other pertinent data from the daily reports. The Project Manager will then check the data, including the proper transcription of the laboratory data onto the figures and tables. Validation of field data will also be conducted by checking the sample locations and depths, sampling methods used, and sample handling procedures. The chain-of-custody form will provide additional information for validation of field procedures.

Validation of the laboratory data will be subcontracted to a data validation subcontractor. They will validate 10 percent of the laboratory results for closure samples against the analytical method criteria. The data validation subcontractor will use the Contract Laboratory Program guidelines in conducting its validation of the laboratory reports and data. These guidelines are presented in USEPA documents titled *National Functional Guidelines for Organic Data Review* (USEPA 1999), and *Draft Laboratory Documentation Requirements for Data Validation* (USEPA 1997) or equivalent update.

The laboratory reports and the data packages are to be evaluated using the laboratory established precision and accuracy limits. For this project, the following quality control criteria will be utilized:

Blank Samples

If an Aroclor is detected in a blank sample at a concentration exceeding the laboratory reporting limit, the test results will be reviewed. If the analyte is found in the blank sample, but not in the field sample, no action is to be taken, and the sample LRL will remain unchanged. Aroclors detected in the field samples that are also detected in the blank sample will be qualified if the sample concentration is less than five times the blank concentration. Corrective action could include re-extracting and re-injecting the batch.

MS Samples

MS samples are analytical samples containing a known concentration of a target analyte that has a known concentration of a target analyte added to it. The MS graphs provide data on the effects of sample preparation and test method for a particular matrix. MS control limits are established for a specific laboratory and its equipment based on historical data. If the relative percent difference (RPD—the difference between the two values divided by the average of the two values) for the MS and MSD samples is outside of MS/MSD control limits and the RPD for the laboratory control standard (LCS) and laboratory control standard duplicate (LCSD) is within the LCS control limits, then the data is to be flagged as suspect due to matrix interference. If the MS/MSD RPD is outside of the MS control limits and the RPD for the LCS/LCSD is outside of its control limits, then the batch is to be re-extracted.

We note that for this project, ERM intends to provide sufficient sample for the laboratory to prepare project-specific MS/MSD samples.

Surrogate Spike Standard

Surrogate recovery control limits are established for a specific laboratory and its equipment based on historical data. If surrogate recoveries for a particular batch of sample are less than the control limits, the batch will be re-extracted and re-injected, and a corrective action report will be written. If surrogate recoveries for a particular batch of sample are greater than the control limits, and PCBs are identified at concentrations exceeding the minimum reporting limit, the batch will be re-extracted and re-injected, and a corrective action report will be written. However, if surrogate recoveries for a particular batch of samples are greater than the control limits, but PCBs are not identified at concentrations exceeding the

minimum reporting limit, no corrective action will be required.

LCS

LCS and LCSD are similar to MS samples except deionized water is used as the matrix. LCS control limits are established for a specific laboratory and its equipment based on historical data. Should matrix interference limit the laboratory's ability to demonstrate precision and accuracy using the MS/MSD data, the LCS/LCSD data will be utilized to demonstrate precision and accuracy. It is ERM's intent to not utilize data that has been rejected by the third party validation firm and/or by ERM's limited Tier I validation. If data is to be qualified, then the reasons for the qualifications and the judgments used for the qualifications are to be documented.

Should issues be discovered by the third party validation that could create an issue with accomplishing the USEPA requested completeness goal of 90 percent, an additional 10 percent of the closure samples analytical data will be sent to the third party validation firm for a Tier IV review. At the request of the USEPA, copies of the raw data for the closure samples will be provided on compact discs to the USEPA.

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5.0 *REPORTING AND SCHEDULE*

5.1 *REPORTING*

After completion of the additional characterization sampling activities, a report will be prepared for the USEPA with the findings. If additional sampling or remediation is required, a table summarizing the cleanup verification sampling will be submitted. Following completion of the closure sampling and testing, and if necessary, decontamination and/or remediation efforts, a final report will be prepared.

The final report will document the investigation, characterization, and remediation activities conducted at the Site. This will include Site diagrams showing the Site, sample locations, and other pertinent data; summaries of the field activities conducted through the time of the report; the results of the laboratory analyses; copies of the laboratory reports and chain-of-custody documents; and the data validation documentation. This and the Certification will be maintained by Mr. Chilcott, as specified in the Certification.

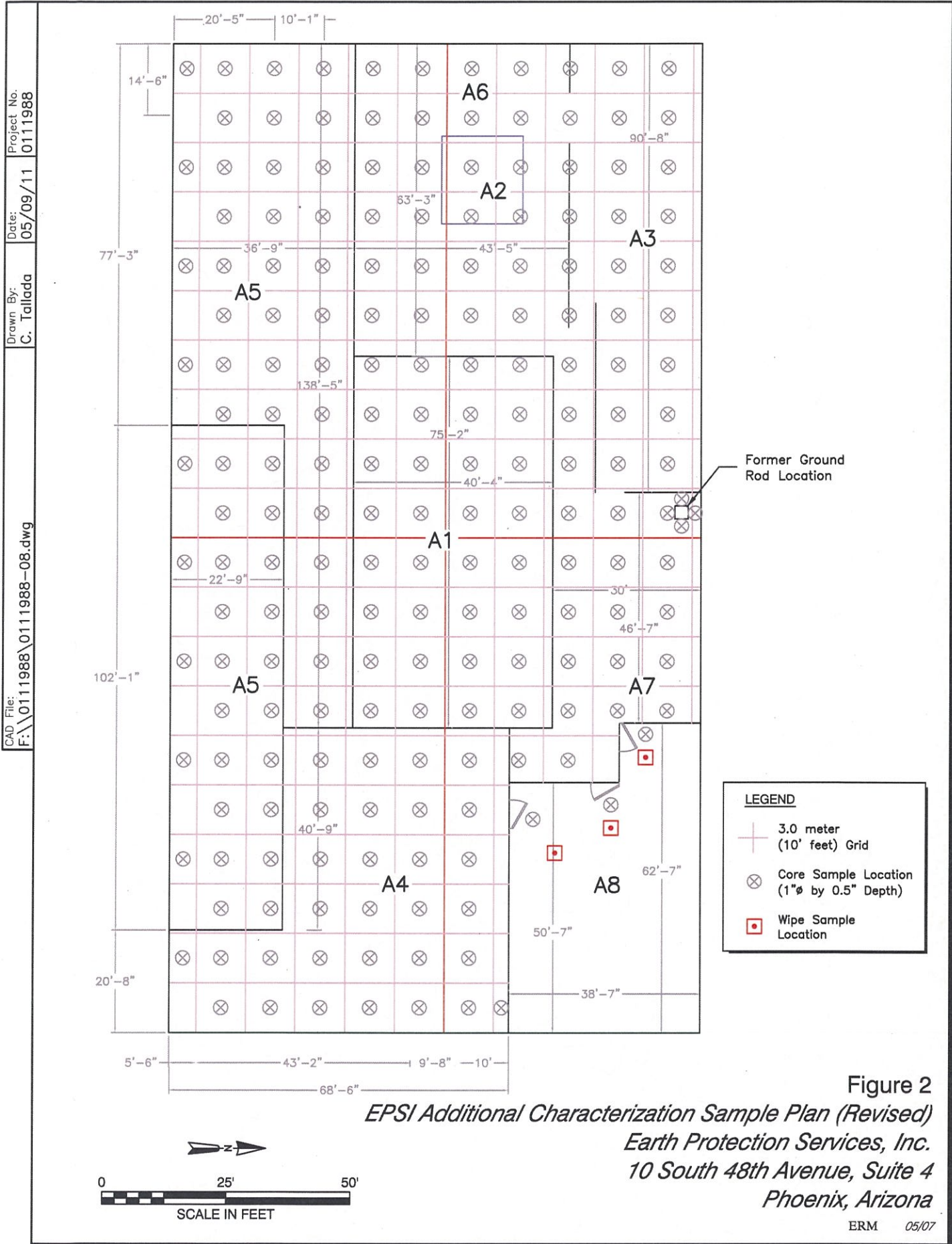
5.2 *SCHEDULE*

EPSI will begin the additional characterization sampling proposed in this plan within 7 days of USEPA approval. Completion of the sampling and analytical work is anticipated to require an additional 21 days. A preliminary letter report will be completed and forwarded to the USEPA Region IX. If additional decontamination is required, this will be scheduled and completed within 21 days. During the decontamination, the table summarizing the cleanup verification sampling plan will be prepared and forwarded to the USEPA.

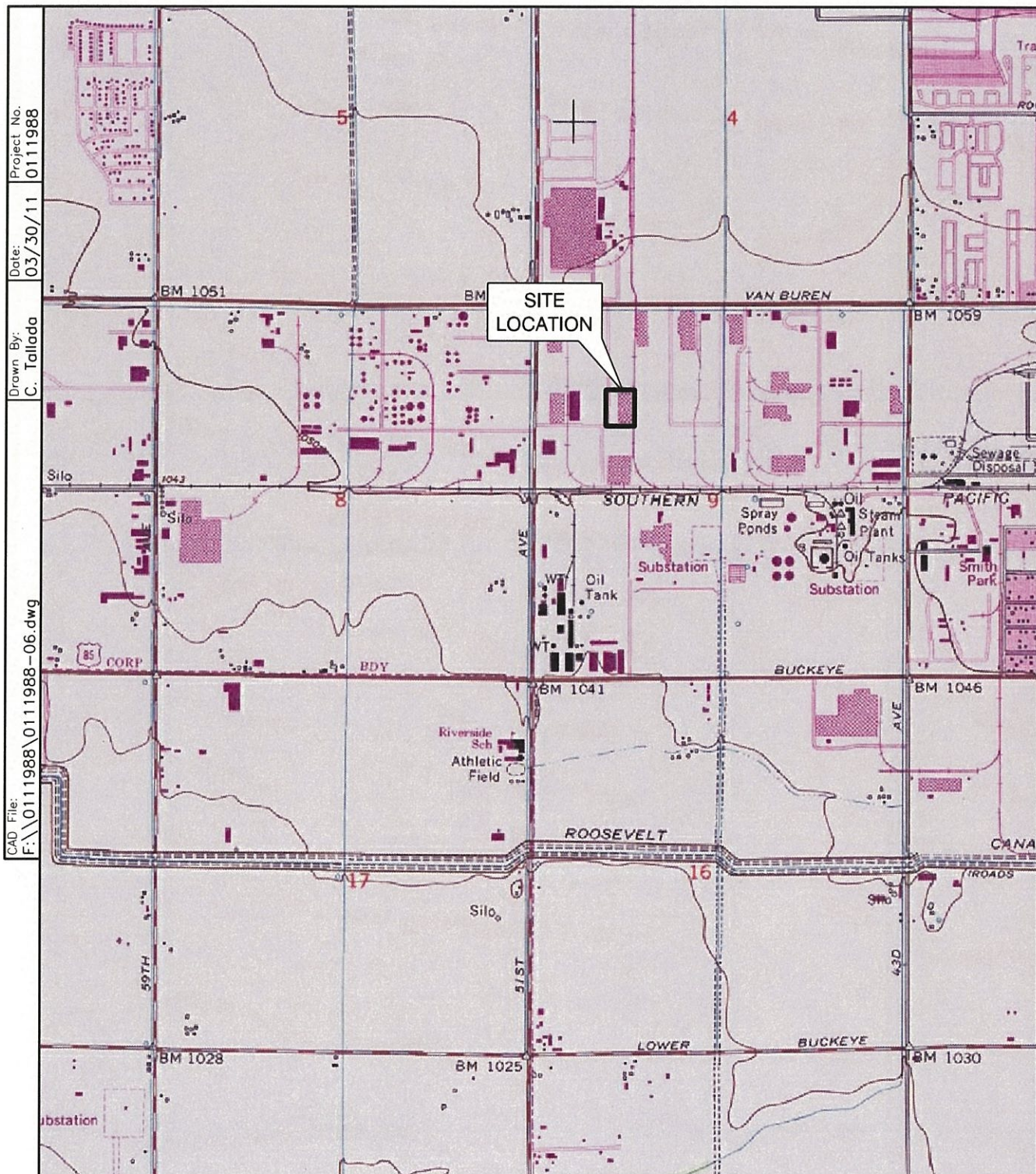
Once samples from an area indicate the area is clean, the area will be marked off so that it will not be further used. For example, if the first round of surface samples from the floor and wall(s) from Area A1 are less than 1 mg/kg total PCB Aroclors, equipment and waste will not be staged in Area A1 and foot traffic will be limited in this area of the Site building.

The order and duration of activities listed above is an initial estimate. This schedule will be revised and adjusted as needed.

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Figures



References:
 U.S.G.S. 7.5 Minute Series (Topographic) Quadrangle,
 (Phoenix, California)
 Map Version: 1981 Current as of: 1978

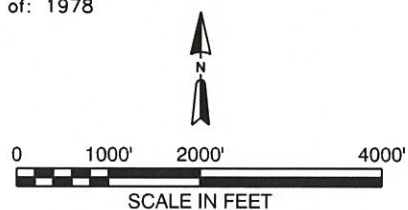
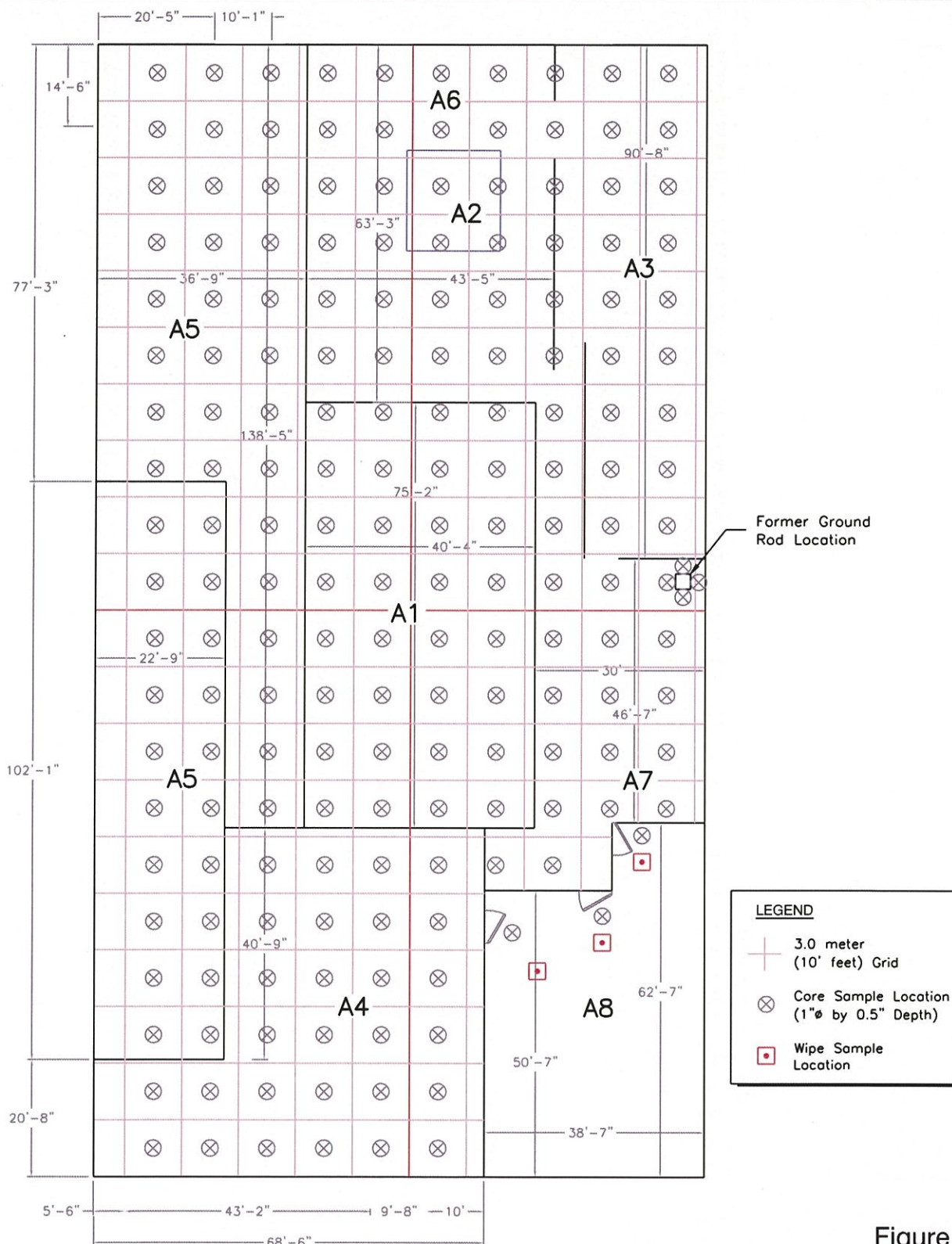


Figure 1
Site Location Map
PCB Sampling Location
Earth Protection Services, Inc.
10 South 48th Avenue, Suite 4
Phoenix, Arizona



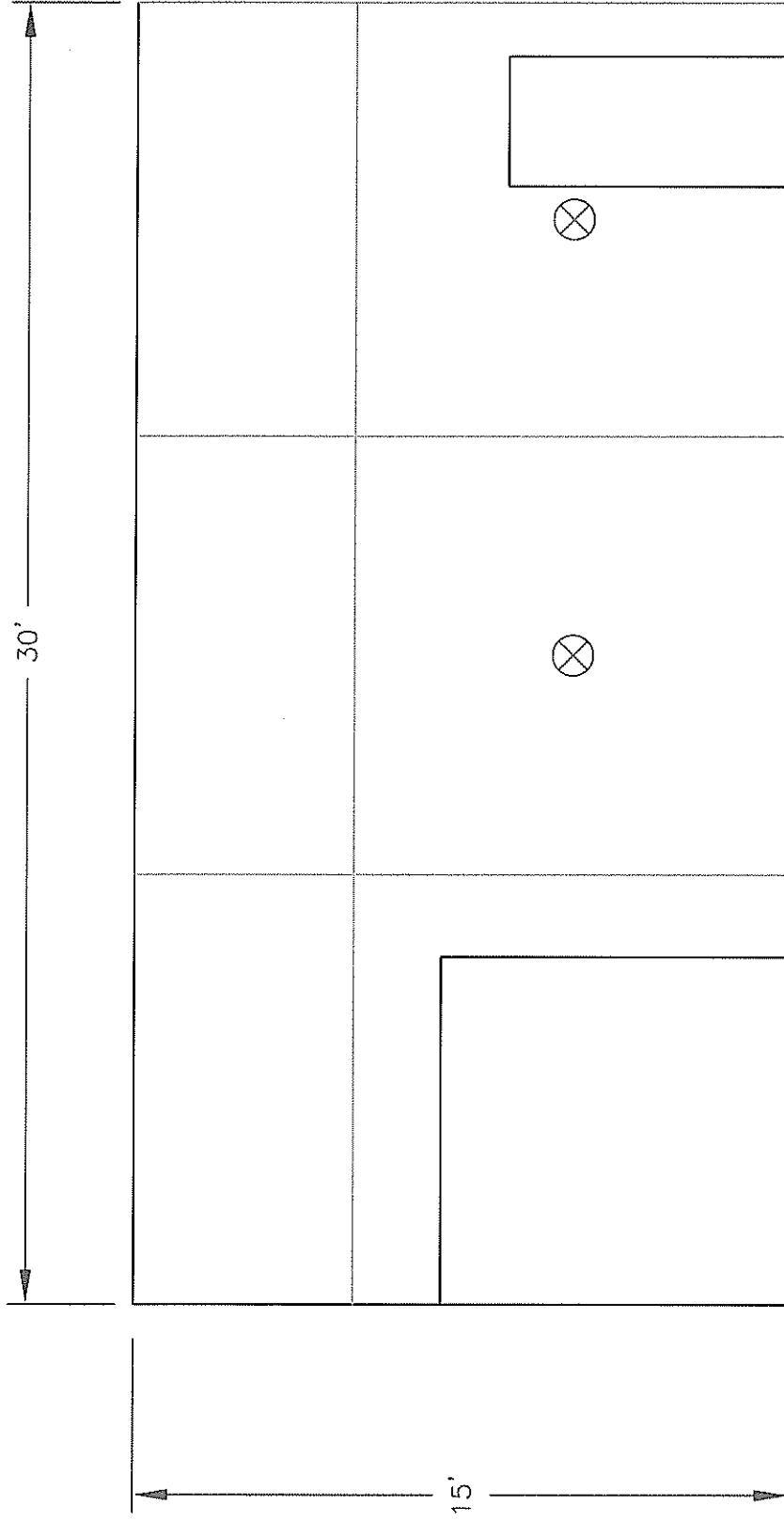
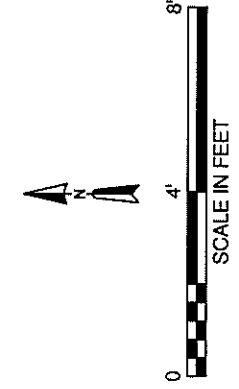


Figure 3
A-3 West Wall Sampling Plan
PCB Sampling Location
Earth Protection Services, Inc.
10 South 48th Avenue, Suite 4
Phoenix, Arizona



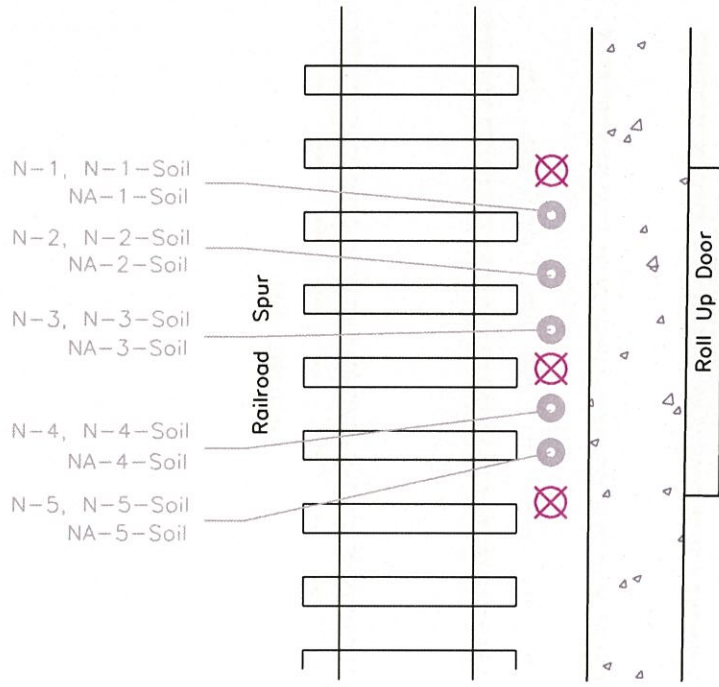
LEGEND	
+	3 meter x 3 meter Grid (10' x 10')
⊗	Core Sample Location (1"Ø)

Project No.
0111988


Date:
03/30/11


Drawn By:
C. Tallada

CAD File:
F:\0111988\0111988-05.dwg

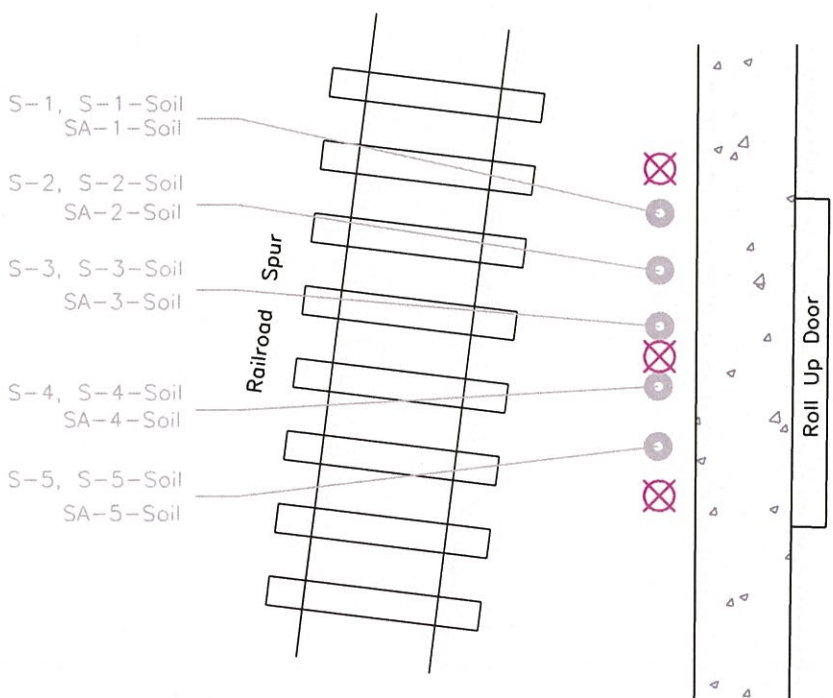


LEGEND

 Bulk Sample Location (1"Ø)

 Soil Sample Location

Detail of North CAFO



Detail of South CAFO

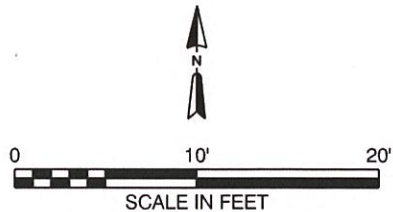
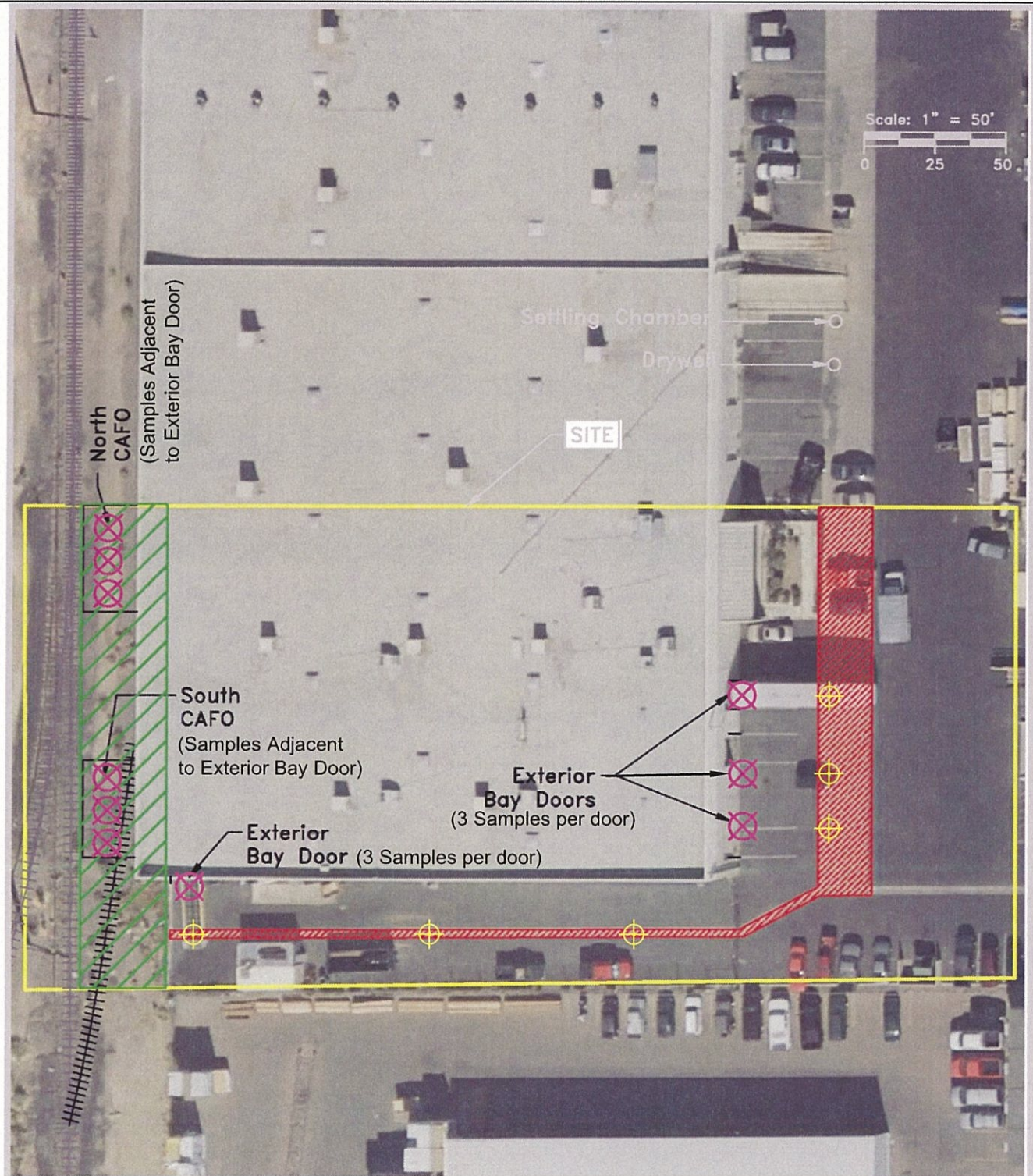


Figure 4
Proposed CAFO Soil Sample Plan
PCB Sampling
Earth Protection Services, Inc.
10 South 48th Avenue, Suite 4
Phoenix, Arizona



LEGEND

- A9**
- A10**
- Bulk Sample Location (1"Ø)
- Core Sample Location (1"Ø by 0.5" Depth)



Figure 5
ESPI Building Exterior Bulk Samples
PCB Sampling
Earth Protection Services, Inc.
10 South 48th Avenue, Suite 4
Phoenix, Arizona

Attachment A
Copy of USEPA Conditional Approval
for Former Earth Protection Services Inc.
Facility, Suite 4, 10 South 48th Avenue,
Phoenix, Arizona, TSCA PCB Cleanup
Under 40 CFR 761.61 (a) and 761.61 (c),
dated March 18, 2011



**UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION IX**

75 Hawthorne Street
San Francisco, CA 94105

Via Electronic Mail and U.S. Postal Service Mail
Certified Mail Receipt No. 7008 1830 0002 6279 5790

March 18, 2011

Gray Boucillon
Cobalt Industrial REIT
5606 North MacArthur Boulevard, Suite 350
Irving, TX 75038

John Chilcott
President, VJ2C, Inc.
2737 E. Arizona Biltmore Circle #4
Phoenix, AZ 85018

Re: Polychlorinated Biphenyls (PCBs), Toxic Substances Control Act - USEPA Conditional Approval Under 40 CFR 761.61(a) and 761.61(c) of "Revised Work Plan Draft Former Earth Protection Services Inc. (EPSI) Facility Phoenix, Arizona" Dated February 7, 2011

Dear Mr. Boucillon and Mr. Chilcott:

On March 7, 2003 the U.S. Environmental Protection Agency's (USEPA's) approval (Toxic Substances Control Act [TSCA] permit) issued to Earth Protection Services, Inc. (EPSI, EPA ID No. AZR000005454) under 40 CFR 761.65 to operate a PCB-commercial storage facility at 10 South 48th Avenue, Suite 4, Phoenix, Arizona, 85043 became effective. Condition D.8, Closure, of the permit required EPSI to close Suite 4 following the closure requirements in the permit and the approved closure plan (Closure Plan). EPSI ceased ballast recycling operations and provided a 60-day closure notification letter on February 3, 2009 in accordance with 40 CFR 761.65 requirements for commercial storers of PCB waste.

This conditional cleanup and disposal approval is being issued by USEPA pursuant to 40 CFR 761.61. In addition, this approval is in accordance with the commercial storer closure requirements in 40 CFR 761.65(e)(7).

Consistent with the above, EPSI/VJC2I submitted for USEPA approval the "Revised Work Plan Draft Former Earth Protection Services Inc. (EPSI) Facility Phoenix, Arizona," (Work Plan) dated February 7, 2011 and prepared by Environmental Resources Management (ERM) for EPSI/VJ2C, Inc. This Work Plan serves as the polychlorinated biphenyls (PCB) cleanup notification (Notification) required by the TSCA regulations in 40 CFR 761.61 for the former EPSI Facility. USEPA received the Notification on February 7, 2011. On February 28, 2011, we found the Notification to be incomplete and notified John Chilcott and Mary Parke (ERM) via e-mail message of this finding. Because of this finding and the subsequent notification to Mr. Chilcott and Ms. Parke, USEPA's 30-day clock set forth in 40 CFR 761.61(a)(3) for responding to the Notification was indefinitely tolled.

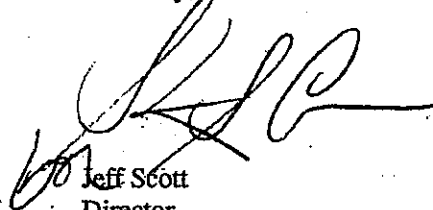
USEPA is approving the Notification under the TSCA regulations in 40 CFR 761.61(a) and 40 CFR 761.61(c). EPSI must implement the Notification as modified by the conditions of approval. In general, the conditions of approval require additional characterization sampling of porous surfaces (e.g., concrete, asphalt), non-porous surfaces (e.g., metal), soil, and PCB-containing dust (including dust from ballast potting material). The approval conditions also require cleanup of porous surfaces and soils to 1 mg/kg PCBs and non-porous surfaces to 10 ug/100 cm² PCBs and these cleanup levels are equivalent to the closure standards in Section D.8.g. (Standards) of EPSI's TSCA permit.

Gray Boucillon (Cobalt Industrial REIT) and John Chilcott (VJC2I)
Re: USEPA Conditional Approval – TSCA PCB Cleanup and Disposal
Former Earth Protection Services Inc. Facility
Date: March 18, 2011

In addition, the approval requires that air sampling be conducted inside and outside Suite 4 after completion of PCB characterization and remediation. USEPA has established a risk-based indoor air standard for PCB Aroclor 1242 expressed as a concentration range equal to 0.021 ug/m^3 (10^{-6} risk level) to 2.1 ug/m^3 (10^{-4} risk level) based on an industrial exposure scenario that must be met in air inside Suite 4. The purpose of this standard is to ensure that post-cleanup PCB indoor air levels are protective of future new occupants of Suite 4. If conducted properly, we anticipate the cleanup of PCBs inside and outside Suite 4 will result in indoor air PCB levels that are within the established air risk-based standard.

We look to EPSI's implementation of the approved PCB Cleanup Notification as modified by USEPA's conditions of approval. Please call Carmen D. Santos at 415.972.3360 if you have any questions concerning this conditional approval.

Sincerely,



Jeff Scott
Director
Waste Management Division

Enclosures (3)

Cc: Mary Parke, ERM
Ivan Lieben, USEPA R9
Arlene Kabei, USEPA R9
Steve Armann, USEPA R9
Carmen Santos, USEPA R9
Edwin Poalinelli, USEPA R9
Patrick Wilson, USEPA R9



**UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION IX
75 Hawthorne Street
San Francisco, CA 94105**

March 18, 2011

**USEPA Conditional Approval for Former Earth Protection Services Inc. Facility
Suite 4, 10 South 48th Avenue, Phoenix, Arizona
TSCA PCB Cleanup Under 40 CFR 761.61(a) and 761.61(c)**

A. Introduction

The U.S. Environmental Protection Agency Region 9 (USEPA) hereby approves with conditions the *"Revised Work Plan Draft Former Earth Protection Services Inc. (EPSI) Facility Suite 4, 10 South 48th Avenue, Phoenix, Arizona,"* dated February 7, 2011 (Notification) for cleanup of polychlorinated biphenyls (PCBs) at the former Earth Protection Services Inc. (EPSI) facility at Suite 4, 10 South 48th Avenue, Phoenix, Arizona. EPSI currently leases the property and Environmental Resources Management (ERM) submitted the Notification to USEPA on behalf of EPSI/VJ2CI. This approval is effective on the date of this enclosure. Section C below contains the conditions of approval.

In this approval, the PCB cleanup site encompasses Suite 4 at 10 South 48th Avenue in Phoenix, Arizona and all outside areas or features immediately surrounding Suite 4 such as soils, sediments, concrete channels, pavement (concrete and/or asphalt) adjacent to Suite 4, railroad spur, and dry well. The term "Facility" is being used interchangeably with "cleanup site."

Any party cleaning up PCBs from soils and structures must do so consistent with the requirements set forth at 40 CFR 761.61. The Toxic Substances Control Act (TSCA) PCB regulations in 40 CFR 761.61 establish PCB cleanup options consisting of self-implementing (40 CFR 761.61(a)), performance-based (40 CFR 761.61(b)), or risk-based (40 CFR 761.61(c)) cleanup alternatives. Depending on site-specific factors, USEPA may approve and require implementation of a PCB cleanup following a hybrid approach that applies requirements from multiple cleanup options.

USEPA is approving the Notification under the TSCA regulatory requirements for PCBs in 40 CFR 761.61(a) and (c). This approval requires additional characterization sampling of soils, asphalt, and dust (on porous and non-porous surfaces) under 40 CFR 761.61(a) and additional characterization sampling of porous surfaces (concrete) under 40 CFR 761.61(c) at the EPSI Facility. In addition, this approval requires cleanup of PCB-contaminated concrete in accordance with 40 CFR 761.61(c) using physical decontamination methods such as the use of abrasives. The cleanup procedure to be used must achieve the cleanup level for concrete equal to or less than 1 mg/kg PCBs in the upper 0.5 to 1 inch layer of the concrete floor and certain walls inside Suite 4.

If PCB-contaminated soils and/or asphalt are present at the Facility, cleanup of these soils and/or asphalt must be achieved via excavation and removal, respectively, followed by offsite disposal in accordance with the requirements in 40 CFR 761.61(a). If PCB-containing dust is present at the Facility, porous and non-porous surfaces must be cleaned up in accordance with 40 CFR 761.61(c).

B. Former EPSI Facility Land Use, Sources of Contamination, Current and Future Use of Former EPSI Facility, and PCB Cleanup

1. **Land Use.** The current and anticipated future land use for the property where EPSI is located is industrial. Suite 4 is vacant and occupies approximately 21,498 square feet (inclusive of former areas A1 through A8).
2. **Sources of PCB Contamination.** According to the Notification, the potting material in PCB light ballasts is a source of PCB contamination at the Facility. PCB contamination has been found at the Facility in both soils and concrete. Aroclor 1242 is the predominant PCB Aroclor detected in soils, dust, and concrete at the Facility.
3. **Current and Future Use of Suite 4.** The use of Suite 4 leased by EPSI will change to other uses; and processing of PCB light ballasts may not continue at Suite 4 by others. Therefore, to allow the continued use of Suite 4 consistent with TSCA use authorizations in 40 CFR 761.30, PCB contaminated building surfaces must be cleaned up in accordance with the requirements in 40 CFR 761.30(u) (Use of decontaminated materials).
4. **PCB Cleanup.** In general, among other requirements, the PCB cleanup at the EPSI Facility includes the following:
 - The approved PCB cleanup level for soils and porous surfaces such as concrete and asphalt is equal to or below 1 mg/kg PCBs.
 - The PCBs cleanup level for non-porous surfaces (e.g., metal) is 10 ug/100 cm² as measured via standard wipe tests.
 - Cleanup of concrete surfaces via approved decontamination methods to achieve the approved cleanup level.
 - Submittal for USEPA approval of a sampling and analysis plan (required in this approval) covering additional soil, concrete, asphalt, dust, and non-porous surfaces (e.g., metal surfaces) characterization sampling and cleanup verification sampling:
 - Additional soil, concrete, asphalt, and metal characterization sampling following the approved sampling and analysis plan. Soil directly beneath asphalt is included in the soils to be sampled.
 - If PCB contaminated soils are present, excavation and offsite disposal of soils contaminated with PCBs above the approved PCB cleanup level.
 - If PCB-contaminated asphalt is present, removal and offsite disposal of the asphalt and excavation of any soils beneath the asphalt that may be contaminated above the 1 mg/kg PCB approved cleanup level for soils and porous surfaces.
 - Soil cleanup verification sampling and analysis.
 - Cleanup of PCB-contaminated concrete to the approved cleanup level and concrete cleanup verification sampling and analysis.
 - Characterization and cleanup verification sampling (via standard wipe tests) and analysis of PCB-containing dust on porous and non-porous surfaces. PCBs must be below 10 ug/100 cm² PCBs.
 - Post PCB cleanup indoor and outdoor air sampling. PCBs must be below the 0.021 ug/m³ (10⁻⁶ risk level) to 2.1 ug/m³ (10⁻⁴ risk level) risk-based standard established in this approval. This air standard is based on an industrial exposure scenario.

USEPA Conditional Approval Under TSCA, 40 CFR 761.61(a) and (c)
Former Earth Protection Services, Inc. Facility, Phoenix, Arizona
Date: March 18, 2011

- PCB Cleanup Completion Report
- Closure Certification for USEPA approval.
- Restrictive covenant recorded in accordance with state law if the approved cleanup levels are not achieved.

C. USEPA's Conditions of Approval

This conditional approval does not relieve the owner from complying with all other applicable federal, state, and local regulations and permits. Given the specific circumstances pertaining to this cleanup, EPSI's compliance with the specified PCB cleanup requirements in 40 CFR 761.61(a) and (c) and the approved Notification as modified by the conditions of approval herein will be deemed to be compliance with the closure requirements in EPSI's TSCA permit issued by USEPA on March 7, 2003.

Departure from the approval conditions herein without prior written permission from USEPA may result in the commencement of proceedings to revoke this approval, and/or an enforcement action. Nothing in this approval bars USEPA from imposing penalties for violations of this approval or for violations of other applicable TSCA PCB requirements or for activities not covered under this approval.

This approval only applies to the former EPSI Facility. USEPA reserves the right to require additional characterization and/or cleanup of PCBs at the EPSI Facility if new information shows that PCBs remain at the Facility above the approved PCB cleanup levels or if PCBs are found at other areas of the Facility.

USEPA is hereby approving the EPSI Notification as modified by the conditions of approval established below. EPSI and the owner of the property must implement the Notification as modified by these conditions.

1. PCB Cleanup Level and Cleanup Verification Sampling Data Comparison to Cleanup Level. The Sampling and Analysis Plan required in Condition 3 of this approval must address Conditions 1.a. through 1.e. below.

- a. Cleanup level for soils, porous surfaces, and non-porous surfaces under 40 CFR 761.61(a).** Under 40 CFR 761.61(a), USEPA is approving a PCB cleanup level for soils and porous surfaces (concrete and asphalt) at the former EPSI Facility equal to or below 1 mg/kg PCBs. Under 40 CFR 761.61(a), USEPA is approving a PCB surface cleanup level of 10 ug/100 cm² PCBs for concrete and non-porous surfaces in connection with PCB-containing dust.

The above cleanup levels are consistent with the closure standards for soils and porous surfaces of 1 mg/kg PCBs and for non-porous surfaces of 10 ug/100 cm² in Section D of EPSI's TSCA permit. EPSI must demonstrate compliance with the soil and asphalt cleanup levels in accordance with the TSCA requirements in 40 CFR 761.61(a)(6). EPSI must demonstrate compliance with the concrete cleanup levels using the procedures in Conditions 1.c. and 1.d. which are developed in accordance with 40 CFR 761.61(c). EPSI must propose in the Sampling Plan required in Condition 3 the method by which it will demonstrate compliance with the surface PCB cleanup level of 10 ug/100 cm².

- b. Demonstration of compliance with cleanup level for soils and asphalt.** When determining if the cleanup level has been met for soils (bulk samples), asphalt (bulk samples), and non-porous surfaces (standard wipe tests) based on analysis results of cleanup verification samples, EPSI shall compare the

analytical results for each soil, asphalt, and non-porous surface cleanup verification sample to the cleanup levels in accordance with 40 CFR 761.61(a)(6). If the PCB concentration of any soil and/or asphalt cleanup verification sample is above the cleanup level, EPSI must continue to clean up (maximum of 3 cleanup rounds) the area directly affected by that sample until the cleanup level is achieved. If the cleanup level is not met, EPSI must confer with USEPA within 5 days of this finding as to next steps.

- c. **Demonstration of compliance with cleanup level for concrete of 1 mg/kg PCBs.** When determining if the cleanup level has been met in concrete based on analysis results of bulk concrete cleanup verification samples, EPSI shall compare the analytical results for each concrete cleanup verification sample to the cleanup level. If the PCB concentration of any concrete cleanup verification sample is above the cleanup level, EPSI must continue to clean up (maximum of 3 cleanup rounds) the area directly affected by that sample until the cleanup level is achieved. If the cleanup level is not achieved after a maximum of three cleanup rounds, EPSI must follow the sealant requirements in Condition 1.d. below.
 - d. **Alternative demonstration of compliance with concrete cleanup level.** Alternatively, EPSI may calculate the distribution-specific 95% confidence limit of the mean (i.e., the exposure-point concentration) of the analysis results for bulk concrete cleanup verification samples using USEPA's ProUCL statistical program and compare that exposure point concentration to the cleanup level. If the exposure point concentration is above the 1 mg/kg PCB cleanup level, EPSI must continue to clean up (a maximum of 3 cleanup rounds) the concrete until the cleanup level is achieved. If the cleanup level is not achieved after a maximum of three cleanup rounds, the concrete must be sealed with a sealant that is effective for PCBs. Condition 1.d. applies only to concrete. Within 14 days after completing the application of the sealant, EPSI must test the sealant using standard wipe tests and PCBs must not be detected above 2 micrograms /100 cm² (ug/100 cm²) PCB Aroclors.
 - e. **Post cleanup PCB indoor air standard under 40 CFR 761.61(c).** Under 40 CFR 761.61(c), USEPA has established an indoor air standard based on an industrial exposure scenario that is a risk-based PCB concentration range of 0.021 ug/m³ (10⁻⁶ risk level) to 2.1 ug/m³ (10⁻⁴ risk level). EPSI must demonstrate the PCB indoor-air concentration do not exceed 2.1 ug/m³ inside Suite 4 after completing removal of PCBs and demonstrating that PCB residual concentrations in soils, porous surfaces, and non-porous surfaces meet the approved cleanup levels.
2. **Certification.** Within 7 calendar days after the date of this approval, EPSI must submit the written, signed certification required in 40 CFR 761.61(a)(3)(i)(E). The certification language should also include the text under the definition of "Certification" in 40 CFR 761.3. The certification must be signed by both the owner of the property and the cleanup party.
 3. **Sampling and Analysis Plan under 40 CFR 761.61(a) and (c).** Within 21 days after the date of this approval, EPSI must submit a Sampling and Analysis Plan (SAP) for USEPA approval that includes the information described below. The SAP is subject to USEPA review and approval and EPSI shall modify the SAP as requested by USEPA. The SAP must address additional soil and asphalt characterization and cleanup verification under 40 CFR 761.61(a), concrete characterization and cleanup verification sampling under 40 CFR 761.61(c), and standard wipe tests for additional concrete and non-porous surfaces.

characterization and cleanup verification in connection with PCB-containing dust under 40 CFR 761.61(c).

- a. **Purpose of sampling and analysis plan.** Data quality objectives for the characterization and cleanup verification sampling.
- b. **Additional characterization sampling.** A table summarizing additional concrete, soil, asphalt, dust, and non-porous surfaces characterization sampling. The summary table must include the media (e.g., soil, concrete) being sampled, type (e.g., discrete), collection method (e.g., wipe samples), and number of samples to be collected for additional characterization, location of samples referencing associated sample identification codes, analytes (e.g., PCB Aroclors), sampling methods, PCB extraction method, laboratory analysis method, laboratory and field quality control (QC) samples, analytical detection limits, and pre-analysis sample extract cleanup methods.
- c. **Concrete cleanup verification sampling.** A table summarizing concrete cleanup verification samples to be collected to demonstrate that after cleanup the PCBs in concrete do not exceed the USEPA approved cleanup level. The type, spacing, and number of samples, associated sample identification codes, location of samples, analytes, sampling methods, laboratory and field QC samples, analytical detection limits, and pre-analysis sample extract cleanup procedures. This table shall be provided within 21 calendar days after implementing decontamination procedures to clean up PCB impacted concrete.
- d. **Soil and asphalt cleanup verification sampling.** If PCB-contaminated soils (including those beneath asphalt and/or concrete) and/or PCB-contaminated asphalt are present at the site above the cleanup level, a table summarizing soil and asphalt cleanup verification samples containing the same information as in the table required in Condition 3.c. above.
- e. **Figures, characterization and cleanup verification samples.** Figures depicting location, spacing, number of samples, and sample identification codes for soil, asphalt, concrete, and dust characterization sampling; and for soil, asphalt, concrete, and dust cleanup verification sampling.
- f. **Sample spacing.** The sample spacing that EPSI will use for collection of concrete core samples, soil, asphalt, and dust at the Facility.
- g. **Indoor and outdoor air sampling.** The SAP must include post PCB cleanup indoor and outdoor air sampling and analysis procedures to demonstrate that PCBs in indoor air do not exceed the indoor air standard. Indoor air sampling will be conducted in Suite 4.
- h. **Quality Control (QC).** A description of quality control (QC) procedures that will be implemented during sample collection and number and type of field QC (e.g., duplicates) samples to be collected for soil, concrete, asphalt, and dust characterization and cleanup verification sampling events. Laboratory QC samples (i.e., surrogate spikes, matrix spikes, equipment blanks) that will be prepared and analyzed by the contracted analytical laboratory.
- i. **Additional SAP requirements.** The SAP must meet the requirements in Conditions 1, 4 through 10, and 12.

4. **Extraction and Analytical Methods.** Under the TSCA PCB regulations the applicant has the option to choose either the Soxhlet extraction method (USEPA Method 3540C) or the Ultrasonic method (USEPA Method 3550C). Our preference is that Soxhlet extraction be used. EPSI must submit the laboratory's Standard Operating Procedure and/or Quality Assurance Project Plan for our review within 21 days after the date of this approval. If necessary, post-extraction and pre-analysis sample cleanup (e.g., USEPA Methods 3665A [sulfuric acid], 3620C [florisil column], 3640A [Gel Permeation Column, GPC]) procedures should be considered if matrix interferences are suspected that could increase analytical method detection limits and compromise comparisons of analytical results to the cleanup levels required in this approval.
5. **Additional Characterization of PCBs in Concrete.** In accordance with the TSCA requirements in 40 CFR 761.61(c) (risk-based PCB cleanup option), USEPA is approving with the conditions below the additional characterization of concrete inside Suite 4.
 - a. EPSI must conduct PCB characterization sampling of the following: (1) entire Facility floor covering the former distinct areas A1 through A7 and the three concrete entry ways inside area A8; and (2) the west wall in area A3.
 - b. EPSI must conduct the required additional characterization following the attached "Standard Operating Procedure for Sampling Porous Surfaces for Polychlorinated Biphenyls (PCBs)," USEPA New England Region 1, July 22, 2008 (SOP).
 - c. In the SAP required in Condition 3, EPSI shall propose the number, spacing, depth, and location of bulk concrete samples that it will collect from the floors and the specified wall and include a figure depicting this information. Concrete sampling locations must encompass and not be limited to locations with visible cracks and stains. Concrete sampling areas should also include concrete immediately adjacent to the grounding rod area.
 - d. Concrete samples must be collected at a depth between 0 to 0.5 inches, not to exceed a maximum depth of 0.5 inches.
 - e. Depending on the PCB analysis results, additional bulk concrete samples must be taken at a depth of 0 to 0.5 inches below surface or deeper depths if necessary to fully characterize the concrete.
 - f. If paint or sealant is present in either the concrete floors or wall, EPSI must propose in the SAP the method to sample the dried paint. Bulk samples of painted concrete must be collected after effective removal of the paint.
6. **Cleanup of Concrete and Cleanup Verification Sampling.** USEPA is approving with the following conditions the cleanup of concrete inside Suite 4 and related cleanup verification sampling under the TSCA PCB regulations in 40 CFR 761.61(c).
 - a. Physical decontamination methods such as scarification or abrasives must be used to clean up the Facility's concrete surfaces impacted by PCBs above 1 mg/kg as demonstrated by analytical results of bulk concrete samples. The required cleanup level is 1 mg/kg.

USEPA Conditional Approval Under TSCA, 40 CFR 761.61(a) and (c)
Former Earth Protection Services, Inc. Facility, Phoenix, Arizona
Date: March 18, 2011

- b. The chosen concrete cleanup method must minimize the generation of dust. Procedures similar to those implemented in lead-based paint or asbestos removal must be implemented for worker protection and minimization of PCB-containing dust.
 - c. EPSI shall propose the number, spacing, and depth of bulk concrete samples that it will collect to verify the cleanup level has been met.
 - d. If based on cleanup verification samples, PCBs remain in the porous surfaces at concentrations above the cleanup level, additional cleanup must be conducted to achieve the cleanup level. Verification samples must be collected between 0 to 0.5 inches below the concrete surface and not to exceed a maximum depth of 0.5 inches.
 - e. If the cleanup level is not met after completing a third round of concrete cleanup, EPSI must follow the requirements in Condition 1.c. or 1.d.
 - f. After cleanup verification samples demonstrate the cleanup level has been achieved, EPSI must remove all dust from the interior surfaces of Suite 4.
- 7. Additional Characterization of PCBs in Soils and Asphalt.** In accordance with the TSCA requirements in 40 CFR 761.61(a), USEPA is approving with the conditions below additional characterization of soils and asphalt (porous surface). Soil sampling was conducted in the past in areas immediately adjacent and in front of the roll-up doors and within the railroad spur and concrete channel in response to USEPA's January 2002 Consent Agreement and Final Order (CAFO). EPSI continued to operate after the CAFO's required sampling and remediation was conducted. Therefore, additional characterization is warranted. In addition, USEPA is requiring that asphalt be characterized for PCBs in the offloading area.
- a. EPSI must propose the location, spacing, depth, and number of soil samples that it will collect in the west side of Suite 4 (including the railroad spur) to demonstrate that post-CAFO EPSI operations have not resulted in recontamination of the CAFO areas (including the railroad spur and concrete channel or gutter) with PCBs.
 - b. EPSI must propose the location, spacing, and number of soil samples that it will collect to demonstrate that PCB contamination above the required cleanup level of 1 mg/kg is not present in soil areas west of Suite 4 not covered by the 2002 CAFO.
 - c. EPSI must propose the location, spacing, and number of soil samples that it will collect in other areas of the Facility. This could include soil areas beneath the concrete slab suspected to be contaminated based on the results of the concrete sampling required in the above Condition 5 and/or beneath asphalt.
 - d. EPSI must propose the location, spacing, and number of asphalt samples that it will collect from the east side of Suite 4 to verify the presence or absence of PCBs.
 - e. EPSI must propose the location, spacing, and number of soil samples that it will collect beneath the locations where it will collect asphalt samples to confirm presence or absence of PCBs in soils beneath the asphalt sampling locations.

- f. The proposals required in Conditions 7.a., 7.b., 7.c., 7.d. and 7.e. must be included in the SAP required in Condition 3 together with figures depicting the number, spacing, depth, and location of samples required in those conditions.
- 8. Cleanup of Soils and Asphalt and Cleanup Verification Sampling.** USEPA is approving cleanup of soils (including any soils beneath asphalt surfaces) and asphalt that may be contaminated with PCBs and related cleanup verification sampling under the TSCA PCB regulations in 40 CFR 761.61(a). Soil cleanup involves excavation of soils contaminated with PCBs above the cleanup level. Asphalt cleanup involves removal of asphalt containing PCBs.
- a. Soils (including PCB-impacted soils beneath asphalt, if any) and asphalt contaminated with PCBs above 1 mg/kg must be removed and disposed offsite in accordance with the requirements in 40 CFR 761.61(a)(5)(i)(B)(2)(ii), (iii), and (iv).
 - b. EPSI shall propose the number, spacing, and depth of soil and asphalt samples that it will collect to verify the cleanup level has been met.
 - c. If based on cleanup verification samples PCBs remain in soil and/or asphalt above the cleanup level, additional cleanup must be conducted to meet the cleanup level.
 - d. If the cleanup level is not met after completing a third round of soil cleanup, EPSI must follow the requirements in Condition 1.
- 9. PCB-Containing Dust on Surfaces inside Suite 4.** EPSI must collect bulk and wipe samples of dust on surfaces inside Suite 4 for PCB analysis before cleanup activities begin to determine if PCB-containing dust is present inside Suite 4. Wipes must be collected in accordance with the standard wipe test procedures in 40 CFR 761.123 and the attached USEPA wipe sampling guidance. Depending on the results of dust samples (bulk and wipes), decontamination of non-porous surfaces and/or removal of other materials within the interior of Suite 4 may be necessary. Non-porous surfaces must be cleaned up to a level equal to or below 10 ug PCBs/100 cm². This cleanup level is consistent with the closure decontamination standard for non-porous surfaces in Condition D.8.g. (Standards) of EPSI's TSCA permit.
- 10. Post Cleanup Air Sampling for PCB Aroclors.** PCB-containing dust may be generated during cleanup of concrete surfaces. EPSI must propose what procedures it will implement to remove dust from the interior of Suite 4 after concrete cleanup verification samples demonstrate the concrete cleanup level has been achieved. To ensure that dust potentially containing PCBs is not present inside Suite 4 post cleanup, EPSI must propose in the SAP the number of indoor and outdoor air samples that it will collect for this purpose.
- USEPA requires that USEPA Method TO-10A or TO-4A (higher air flow rate) be used for collection and analysis of the indoor and outdoor air samples.
- 11. Deed Notice, Sealant Inspection, Maintenance, and Repair Plan (Sealant Plan).** If a sealant has to be applied to cleaned-up concrete surfaces because the cleanup level was not achieved, EPSI must record a deed notice in accordance with state law that includes the USEPA-approved EPSI sealant inspection,

maintenance, and repair plan (Sealant Plan). The deed notice must include the Sealant Plan and the information required in 40 CFR 761.61(a)(8).

12. **Decontamination of Movable Equipment, Tools, and Sampling Equipment Contaminated by PCBs.** Equipment not covered in the USEPA Region 1 SOP must be decontaminated following the requirements in 40 CFR 761.79(c)(2).
13. **PCB Waste Disposal, Decontamination Residues, and Cleanup Wastes.** Decontamination residues (e.g., spent abrasives) and cleanup wastes must be disposed based on their original PCB concentration in accordance with 40 CFR 761.79(g)(2), (g)(6), and 40 CFR 761.61(a)(5), (a)(5)(i), (a)(5)(iii), and (a)(5)(v). Concrete and/or other porous surfaces(s) contaminated with PCBs above the approved PCB cleanup level must be disposed as bulk PCB remediation waste in accordance with the requirements in 40 CFR 761.61(a)(5)(i)(B)(2)(ii), (a)(5)(v)(A), and (a)(5)(i)(B)(2)(iii). Disposal of all wastes (e.g., personal protective equipment, soils, concrete) generated during cleanup of PCBs must be in compliance with all applicable federal, state, and local regulations.
14. **Request for Additional Information.** EPSI has conducted sampling and removal of materials within certain areas of the Facility and has used the results of these activities in determining that PCBs are not present at certain areas of the Facility above the cleanup levels (i.e., the closure standards). Within 21 days after the date of this approval, EPSI must submit for USEPA review a Status Report that provides the information requested in Conditions 14.a. through 14.c.
 - a. **Area 3 "Black Wall."** USEPA is concerned about the wall in the Process Area that is painted a dark color. This wall was adjacent to the work table and its surface has a high potential to be contaminated with PCBs. During a conference call with USEPA, ERM (EPSI's contractor) stated that the "Black Wall" no longer exists. EPSI must provide a summary of the removal activities, explain if bulk samples were collected from the wall for PCB analysis and if the paint was removed from the wall before bulk sample collection, and if the paint was sampled and analyzed for PCBs. In addition, EPSI must provide photos documenting the complete removal of the "Black Wall." EPSI must include laboratory analytical reports associated with samples collected from the "Black Wall" and table summarizing the laboratory analytical results. EPSI shall also describe how it disposed of the "Black Wall."
 - b. **Grounding rod area.** EPSI must provide a detailed summary of all characterization sampling, cleanup and removal activities, and cleanup verification sampling conducted in the grounding rod area. This summary must include a figure depicting sampling locations, sample analytical results, and removal activities. The summary must include a table of sample results, photo documentation, and copies of laboratory analytical reports.
 - c. **Drainage dry well.** EPSI must provide a summary of sampling activities associated with the dry well and the concrete channel (or gutter) leading to the dry well (see Condition 14.d.). EPSI must submit documentation and an explanation supporting its statement that the dry well has never been impacted by PCBs. Any summary of laboratory analytical results and copies of laboratory analytical reports must be submitted.

- d. **Outdoor concrete channel (drainage) areas.** EPSI must provide a detailed summary of all samples collected in outdoor concrete channel (drainage) areas (37 core samples collected from the drainage areas south of Suite 4 and 37 core samples from the east of Suite 4). The summary must include the sampling protocol used, figure depicting sampling locations and sample analytical results; a table summarizing sample analytical results; and explanation supporting that PCBs were not detected in the channel above the cleanup level (i.e., closure standard).

15. **PCB Cleanup Completion Report and Closure Certification.** Within 60 days after EPSI demonstrates that residual PCBs in soils, asphalt, sediments, concrete, and non-porous surfaces are equal to or below the cleanup level, the interior of Suite 4 does not contain PCB-containing dust, and the post-cleanup indoor air samples are below the risk-based air standard for Suite 4, EPSI must submit a PCB Cleanup Completion Report (PCB Cleanup Report) for USEPA approval (under 40 CFR 761.61(c)). This report must comply with all the requirements in Condition D.8.f. of EPSI's TSCA permit and all reporting requirements in 40 CFR 761.61(a)(9) and 761.125(c)(5) not specifically required in Condition D.8.f. of the TSCA permit. The PCB Cleanup Report must provide all relevant sampling and analysis data and justifications demonstrating that EPSI achieved the USEPA approved PCB cleanup levels and post-cleanup risk-based air standards at the Facility, and that it met the conditions of approval. In addition, the PCB Cleanup Report must include as an attachment the USEPA-approved Status Report required in Condition 14. The Status Report required in that Condition is subject to USEPA approval.

D. Not Covered by this Approval

1. **Health and Safety Plan.** This approval does not cover approval of any Health and Safety Plan(s) that may be referenced in the Notification or other documents incorporated into the Notification by reference.



**UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION IX**

75 Hawthorne Street
San Francisco, CA 94105

Via Electronic Mail and U.S. Postal Service Mail
Certified Mail Receipt No. 7008 1830 0002 6279 5790

March 18, 2011

Gray Boucillon
Cobalt Industrial REIT
5606 North MacArthur Boulevard, Suite 350
Irving, TX 75038

John Chilcott
President, VJ2C, Inc.
2737 E. Arizona Biltmore Circle #4
Phoenix, AZ 85018

Re: Polychlorinated Biphenyls (PCBs), Toxic Substances Control Act - USEPA Conditional Approval Under 40 CFR 761.61(a) and 761.61(c) of "Revised Work Plan Draft Former Earth Protection Services Inc. (EPSI) Facility Phoenix, Arizona" Dated February 7, 2011

Dear Mr. Boucillon and Mr. Chilcott:

On March 7, 2003 the U.S. Environmental Protection Agency's (USEPA's) approval (Toxic Substances Control Act [TSCA] permit) issued to Earth Protection Services, Inc. (EPSI, EPA ID No. AZR000005454) under 40 CFR 761.65 to operate a PCB commercial storage facility at 10 South 48th Avenue, Suite 4, Phoenix, Arizona, 85043 became effective. Condition D.8, Closure, of the permit required EPSI to close Suite 4 following the closure requirements in the permit and the approved closure plan (Closure Plan). EPSI ceased ballast recycling operations and provided a 60-day closure notification letter on February 3, 2009 in accordance with 40 CFR 761.65 requirements for commercial storers of PCB waste.

This conditional cleanup and disposal approval is being issued by USEPA pursuant to 40 CFR 761.61. In addition, this approval is in accordance with the commercial storer closure requirements in 40 CFR 761.65(e)(7).

Consistent with the above, EPSI/VJC2I submitted for USEPA approval the "Revised Work Plan Draft Former Earth Protection Services Inc. (EPSI) Facility Phoenix, Arizona," (Work Plan) dated February 7, 2011 and prepared by Environmental Resources Management (ERM) for EPSI/VJ2C, Inc. This Work Plan serves as the polychlorinated biphenyls (PCB) cleanup notification (Notification) required by the TSCA regulations in 40 CFR 761.61 for the former EPSI Facility. USEPA received the Notification on February 7, 2011. On February 28, 2011, we found the Notification to be incomplete and notified John Chilcott and Mary Parke (ERM) via e-mail message of this finding. Because of this finding and the subsequent notification to Mr. Chilcott and Ms. Parke, USEPA's 30-day clock set forth in 40 CFR 761.61(a)(3) for responding to the Notification was indefinitely tolled.

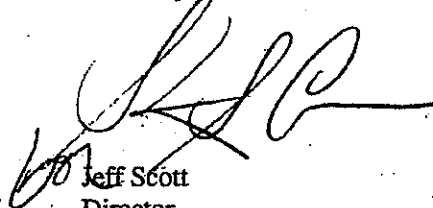
USEPA is approving the Notification under the TSCA regulations in 40 CFR 761.61(a) and 40 CFR 761.61(c). EPSI must implement the Notification as modified by the conditions of approval. In general, the conditions of approval require additional characterization sampling of porous surfaces (e.g., concrete, asphalt), non-porous surfaces (e.g., metal), soil, and PCB-containing dust (including dust from ballast potting material). The approval conditions also require cleanup of porous surfaces and soils to 1 mg/kg PCBs and non-porous surfaces to 10 ug/100 cm² PCBs and these cleanup levels are equivalent to the closure standards in Section D.8.g. (Standards) of EPSI's TSCA permit.

Gray Boucillon (Cobalt Industrial REIT) and John Chilcott (VJC2I)
Re: USEPA Conditional Approval – TSCA PCB Cleanup and Disposal
Former Earth Protection Services Inc. Facility
Date: March 18, 2011

In addition, the approval requires that air sampling be conducted inside and outside Suite 4 after completion of PCB characterization and remediation. USEPA has established a risk-based indoor air standard for PCB Aroclor 1242 expressed as a concentration range equal to 0.021 ug/m^3 (10^{-6} risk level) to 2.1 ug/m^3 (10^{-4} risk level) based on an industrial exposure scenario that must be met in air inside Suite 4. The purpose of this standard is to ensure that post-cleanup PCB indoor air levels are protective of future new occupants of Suite 4. If conducted properly, we anticipate the cleanup of PCBs inside and outside Suite 4 will result in indoor air PCB levels that are within the established air risk-based standard.

We look to EPSI's implementation of the approved PCB Cleanup Notification as modified by USEPA's conditions of approval. Please call Carmen D. Santos at 415.972.3360 if you have any questions concerning this conditional approval.

Sincerely,



Jeff Scott
Director
Waste Management Division

Enclosures (3)

Cc: Mary Parke, ERM
Ivan Lieben, USEPA R9
Arlene Kabei, USEPA R9
Steve Armann, USEPA R9
Carmen Santos, USEPA R9
Edwin Poalinelli, USEPA R9
Patrick Wilson, USEPA R9

Attachment B
Accutest QA Manual

Compound List Report

Product: P8082PCB Polychlorinated Biphenyls

Matrix: SO Solid

Jan 04, 2011 05:37 pm

Method List: P8082 SO

Method Ref: SW846 8082

LC718

Report List: PCB ALL

PCB List

LF2924

RL/MDL Factor: 1

Compound	CAS No.	RL	MDL	Units	Control Limits (%) Rev: 03/26/10			
					MS/MSD	RPD	BS	DUP
Aroclor 1016	12674-11-2	100	17 ug/kg	40-145	40	40-145		40
Aroclor 1221	11104-28-2	100	50 ug/kg	40-145	40	40-145		40
Aroclor 1232	11141-16-5	100	50 ug/kg	40-145	40	40-145		40
Aroclor 1242	53469-21-9	100	50 ug/kg	40-145	40	40-145		40
Aroclor 1248	12672-29-6	100	50 ug/kg	40-145	40	40-145		40
Aroclor 1254	11097-69-1	100	50 ug/kg	40-145	40	40-145		40
Aroclor 1260	11096-82-5	100	20 ug/kg	40-145	40	40-145		40
Tetrachloro-m-xylene	877-09-8			Surrogate Limits:	45-108			
Decachlorobiphenyl	2051-24-3			Surrogate Limits:	54-121			

7 compounds and 2 surrogates reported in list PCB

Compound List Report

Product: P8082PCB Polychlorinated Biphenyls
Matrix: SO Solid

3/21/2011 14:52

Method List: P8082 SO
Report List: PCB ALL
RL/MDL Factor: 0.33

Method Ref: SW846 8082
PCB List

LC718
LF2924

Compound	CAS No.	RL	MDL	Units	Control Limits (%) Rev: 03/26/10		
					MS/MSD	RPD	BS
Aroclor 1016	12674-11-2	33		5.6 ug/kg	40-145		40 40-145
Aroclor 1221	11104-28-2	33		17 ug/kg	40-145		40 40-145
Aroclor 1232	11141-16-5	33		17 ug/kg	40-145		40 40-145
Aroclor 1242	53469-21-9	33		17 ug/kg	40-145		40 40-145
Aroclor 1248	12672-29-6	33		17 ug/kg	40-145		40 40-145
Aroclor 1254	11097-69-1	33		17 ug/kg	40-145		40 40-145
Aroclor 1260	11096-82-5	33		6.6 ug/kg	40-145		40 40-145
Tetrachloro-m-xylene	877-09-8						45-108
Decachlorobiphenyl	2051-24-3						54-121

7 compounds and 2 surrogates reported in list PCB

Surrogate Limits:

Surrogate Limits:

Compound List Report

Product: P8082PCB Polychlorinated Biphenyls

Matrix: WIPE Wipe

#####

Method List: P8082 W Method Ref: SW846 8082

LC749

Report List: PCB ALL PCB List

LF2924

RL/MDL Factor: 0.01

Compound CAS No.	RL	MDL	Units	Control Limits (%)		BS	DUP
				MS/MSD	RPD		
Aroclor 101 12674-11-2	1	0.5 ug/wipe	40-145			40 40-145	40
Aroclor 122 11104-28-2	1	0.5 ug/wipe	40-145			40 40-145	40
Aroclor 123 11141-16-5	1	0.5 ug/wipe	40-145			40 40-145	40
Aroclor 124 53469-21-5	1	0.5 ug/wipe	40-145			40 40-145	40
Aroclor 124 12672-29-5	1	0.5 ug/wipe	40-145			40 40-145	40
Aroclor 125 11097-69-1	1	0.5 ug/wipe	40-145			40 40-145	40
Aroclor 125 11096-82-5	1	0.5 ug/wipe	40-145			40 40-145	40

Tetrachloro 877-09-8

Surrogate Limits:

58-130

Decachloro 2051-24-3

Surrogate Limits:

58-130


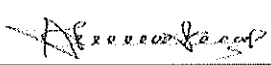
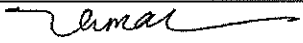
7 compounds and 2 surrogates reported in list PCB

Quality Systems Manual

Revision VIII: February 21, 2011

Effective Date: February 21, 2011

Uncontrolled Copy

Approved by: (Signature/Date)	
Laurie Glantz-Murphy Laboratory/Technical Director	
Guergana Gueorguieva Quality Assurance Officer	
Mai Tran SVOC Supervisor	

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Introduction

The Accutest Laboratories – Northern California Quality Assurance System, detailed in this plan, has been designed to meet the quality program requirements of the National Environmental Laboratory Accreditation Conference (NELAC), ISO Guide 17025, ISO Guide 17011 and other National environmental monitoring programs. The plan establishes the framework for documenting the requirements of the quality processes regularly practiced by the Laboratory. The Quality Assurance Director is responsible for changes to the Quality Assurance Program, which is appended to the Quality System Manual (QSM) during the annual program review. The plan is also reviewed annually for compliance purposes by the Company President and Laboratory Director and edited if necessary. Changes that are incorporated into the plan are itemized in a summary of changes following the introduction. Plan changes are communicated to the general staff in a meeting conducted by the Director of Quality Assurance following the plan's approval.

The Accutest plan is supported by standard operating procedures (SOPs), which provide specific operational instructions on the execution of each quality element and assure that compliance with the requirements of the plan are achieved. Accutest employees are responsible for knowing the requirements of the SOPs and applying them in the daily execution of their duties. These documents are updated as changes occur and the staff is trained to apply the changes.

At Accutest, we believe that satisfying client requirements and providing a product that meets or exceeds the standards of the industry is the key to a good business relationship. However, client satisfaction cannot be guaranteed unless there is a system that assures the product consistently meets its design requirements and is adequately documented to assure that all procedural steps are executed, properly documented and traceable.

This plan has been designed to assure that this goal is consistently achieved and the Accutest product withstands the rigors of scrutiny that are routinely applied to analytical data and the processes that support its generation.

Summary of Changes

Accutest Laboratories Quality System Manual – February 2011

Section	Description
	SOP Number, Updated Revision Number and Date
	Original issue due to Sale February 2008
	QC008_2 - Revision II May 1, 2008 Changes
Appendix II	Updated SOP List
2.3	Assigned Deputies to Laboratory Director and Quality Assurance Officer
	QC008_3 - Revision III March 6, 2009 Changes
Appendix IV	Updated Equipment list – add ICS2000
Sect. 2	Updated Organizational Chart
	QC008_4 - Revision IV September 7, 2009 Changes
Cover page	Change address
Section 1.1	Added including the DoD QSM
Section 2.1	Change Santa Clara to San Jose
Section 2.1	Added ANC is a permanent location and not a mobile of temporary facility
Section 2.3	Added the laboratory designate another full-time staff member meeting the qualifications of the technical director to temporarily perform this function when the technical director is absent for a period of time exceeding 15 days. If this absence exceeds 65 consecutive days, the primary accrediting authority shall be notified in writing
Section 6.1	Standard Operating Procedures (SOP), deleted in the master SOP binder
Section 9.8	Subcontract Laboratory Evaluation-added All subcontracted items for testing covered under DoD will only be submitted to DoD-ELAP laboratories
Section 15.1	Procedure-inserted If no systematic defects are present and the proposed resolutions is sufficient, QA will close the complaint/inquiry with a No Further Action is necessary
	QC008_5- Revision V December 31, 2009 changes
Cover, 2.2,2.3, 3.1,4.1,5.1	Changing the title and responsibilities from "Laboratory Director" to "Laboratory/Technical Director".
5.1	Signatory Hierarchy Changed
	QC008_6- Revision VI February 15, 2010 changes
5.1	Signatory Hierarchy Changed
2.1,2.2	Added Accutest Laboratories-Mountain States to the Network.
10.4	Added text to define a Second Source Standard.
	QC008_7- Revision VII October 20, 2010 changes
Section 2.3	Accutest Northern California – Organization Chart
Appendix II	Standard Operational Procedures Directory
Appendix IV	Laboratory Equipment
	QC008_8-Revision VIII February 18, 2011 changes
Section 2.3	Accutest Northern California – Organization Chart
Section 16.3	Added Data Inquiry Program
Appendix II	Standard Operating Procedures
Appendix IV	Laboratory Equipment –added GCMS Q, GCMS R and Thermo Fisher ICP

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1.0 QUALITY POLICY

1.1 Accutest Mission:

Accutest Laboratories provides analytical services to commercial and government clients in support of environmental monitoring and remedial activities as requested. The Laboratory's mission is dedicated to providing reliable data that satisfies client's requirements as explained in the following:

"Provide easy access, high quality, analytical support to commercial and government clients including those following the DoD QSM, which meets or exceeds data quality objectives and provides them with the data needed to satisfy regulatory requirements and/or make confident decisions on the effectiveness of remedial activities."

These services are provided impartially and are not influenced by undue commercial or financial pressures which might impact the staff's technical judgment. Coincidentally, Accutest does not engage in activities that endanger the trust in our independent judgment and integrity in relation to the testing activities performed.

1.2 Policy Statement:

The management and staff of Accutest Laboratories share the responsibility for product quality. Accordingly, ANC's quality assurance program is designed to assure that all processes and procedures, which are components of environmental data production, meet established industry requirements, are adequately documented from a procedural and data traceability perspective, and are consistently executed by the staff. It also assures that analytical data of known quality, meeting the quality objectives of the analytical method in use and the data user's requirements, is consistently produced in the laboratory. This assurance enables the data user to make rational, confident, cost-effective decisions on the assessment and resolution of environmental issues.

The laboratory Quality System also provides the management staff with data quality and operational feedback information. This enables them to determine if the laboratory is achieving the established quality and operational standards, which are dictated by the client or established by regulation. The information provided to management, through the QA program, is used to assess operational performance from a quality perspective and to perform corrective action as necessary.

All employees of Accutest Laboratories participating in environmental testing receive quality system training and are responsible for knowing and complying with the system requirements. The entire staff shares ANC's commitment to good professional practice.


Vincent J. Pugliese, President

January 25, 2011

Date

2.0 ORGANIZATION

- 2.1 **Organizational Entity.** Accutest Laboratories is a privately held, independent testing laboratory founded in 1956 and registered as a New Jersey Corporation. The headquarters are located in Dayton, New Jersey where it has conducted business since 1987. Satellite laboratories are maintained in Marlborough, Massachusetts; Orlando, Florida; Houston, Texas; San Jose, California and Wheatridge, Colorado.

Accutest Northern California is a permanent location and not a mobile or temporary facility

2.2 **Management Responsibilities**

Requirement: Each laboratory facility has an established chain of command. The duties and responsibilities of the management staff are linked to the President/CEO of Accutest Laboratories who establishes the agenda for all company activities.

President/CEO. Primary responsibility for all operations and business activities. Delegates authority to laboratory directors, general managers, and the quality assurance director to conduct day to day operations and execute quality assurance duties. Each of the five operational entities (New Jersey, Florida, Massachusetts, California, Texas and Colorado) report to the President/CEO.

Vice President Operations (Corporate). Delegates responsibility for laboratory operations including technical aspects of production activities and associated logistical procedures to the respective Laboratory and/or Technical Directors. VP of Operations reports directly to the President/CEO.

Laboratory Director (Local). Executes day to day responsibility for laboratory operations including technical aspects of production activities and associated logistical procedures. Reports directly to the Vice President of Operations.

Corporate Quality Assurance Director (Corporate). Responsible for design, implementation support, training, and monitoring of the quality system. Identifies product, process, or operational defects using statistical monitoring tools and processes audits for elimination via corrective action. Empowered with the authority to halt production if warranted by quality problems. Monitors implemented corrective actions for compliance.

Quality Assurance Officer (Local). Responsible for design support, implementation support, and monitoring support of the quality system. Training personnel in various aspects of quality system. Conducts audits and product reviews to identify product, process, or operational defects using statistical monitoring tools and processes audits for elimination via corrective action. Empowered with the authority to halt production if warranted by quality problems. Monitors implemented corrective actions for compliance.

Department Supervisors. Executes day to day responsibility for specific laboratory areas including technical aspects of production activities and associated logistical procedures. Direct report to the laboratory director.

Bench Analysts. Responsible for applying the requirements of the Quality Program to the analyses they perform, evaluating QC data and initiating corrective action for quality control deficiencies within their control. Implements global corrective action as directed by superiors.

2.3 Chain of Command

The laboratory designates another full-time staff member meeting the qualifications of the technical director(s) to temporarily perform this function when the technical director(s) is absent for a period of time exceeding 15 consecutive calendar days. If this absence exceeds 65 consecutive calendar days, the primary accrediting authority shall be notified in writing. The responsibility for managing all aspects of ANC's operation is delegated to specific local individuals, who have been assigned the authority to act in the absence of the senior staff. Deputies will perform duties in the absence of their designees if the absence is greater than three weeks. These individuals are identified in the following Chain of Command:

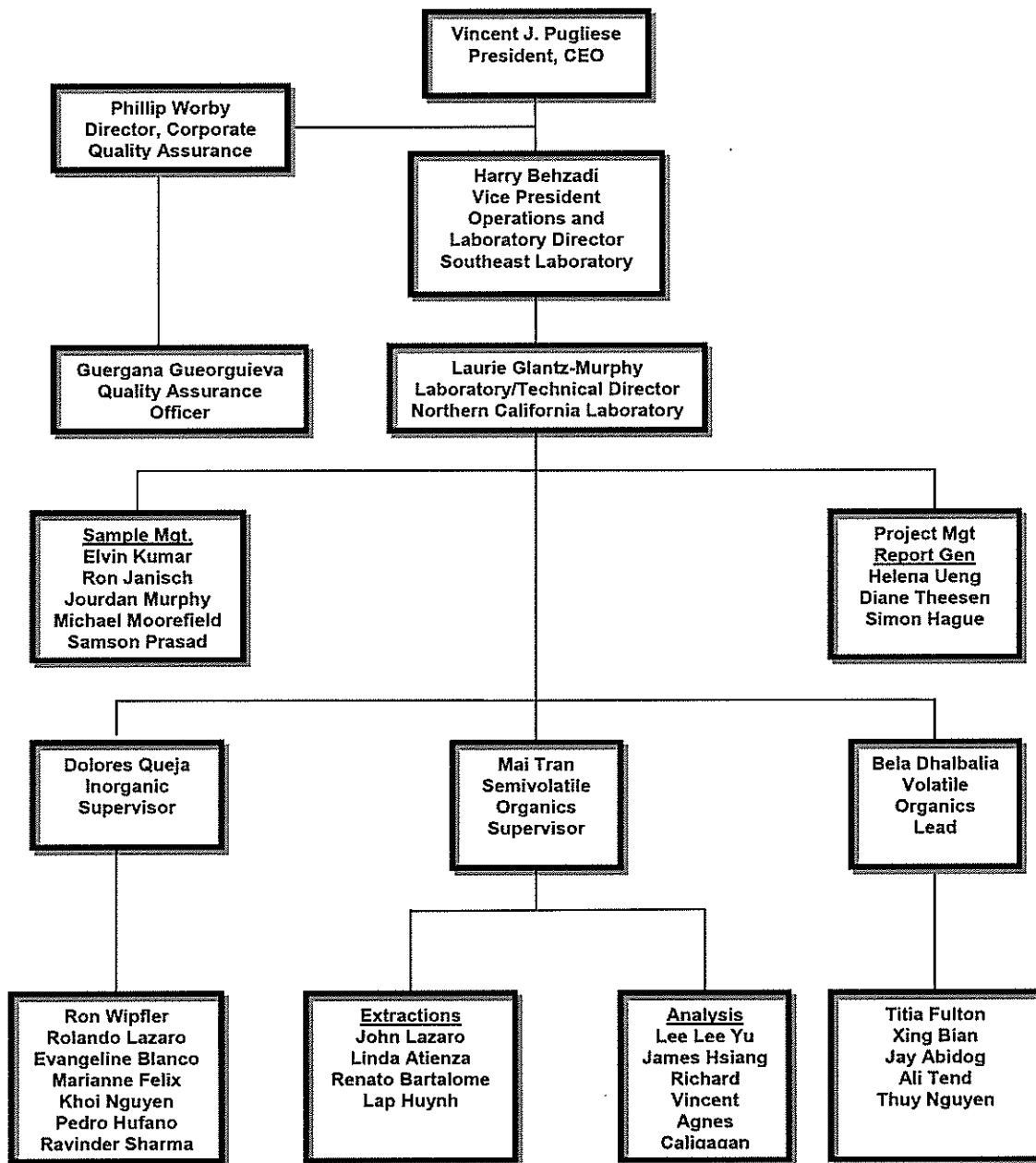
Laurie Glantz-Murphy; Laboratory/Technical Director

Deputy: Mai Tran

Guergana Gueorgueva; Quality Assurance Officer

Deputy: Dolores Queja

Accutest Northern California - Organization



3.0 QUALITY RESPONSIBILITIES OF THE MANAGEMENT TEAM

- 3.1 **Requirement:** Each member of the management team has a defined responsibility for the Quality System. System implementation and operation is designated as an operational management responsibility. System design and implementation is designated as a Quality Assurance Responsibility.

President/CEO. Primary responsibility for all quality activities. Delegates program responsibility to the Quality Assurance Director. Serves as the primary alternate in the absence of the Quality Assurance Director. Has the ultimate responsibility for implementation of the Quality System.

Vice President Operations (Corporate). Responsible for implementing and operating the Quality System in all laboratory areas. Responsible for the design and implementation of corrective action for defective processes. Has the authority to delegate Quality System implementation responsibilities.

Quality Assurance Director (Corporate) Responsible for design, implementation support, training, and monitoring of the quality system. Identifies product, process, or operational defects using statistical monitoring tools and processes audits for elimination via corrective action. Empowered with the authority to halt production if quality issues warrant immediate action. Monitors implemented corrective actions for compliance.

Quality Assurance Officer (Local). Responsible for design, implementation support, training, and monitoring support for the quality system. Conducts audits and product reviews to identify product, process, or operational defects using statistical monitoring tools and processes audits for elimination via corrective action. Provides monitors support for implemented corrective actions for compliance.

Laboratory/Technical Director. Responsible for implementing, operating, and technical reviewing of the Quality System in all the local laboratory areas. Responsible for design and implementation of the corrective action process.

Department Supervisors. Responsible for applying the requirements of the Quality System in their section and assuring subordinate supervisors and staff apply all system requirements. Initiates, designs, documents, and implements corrective action for quality deficiencies.

Bench Analysts. Responsible for applying the requirements of the Quality System to the analyses they perform, evaluating QC data and initiating corrective action for quality control deficiencies within their control. Implements global corrective action as directed by superiors.

- 3.2 **Program Authority.** Authority for program implementation originates with the President/CEO who bears the ultimate responsibility for system design, implementation, and enforcement of requirements. This authority and responsibility is delegated to the Director of Quality Assurance who performs quality functions independently without the encumbrances or biases associated with operational or production responsibilities to ensure an honest, independent assessment of quality issues.
- 3.3 **Data Integrity Policy.** The Accutest Data Integrity Policy reflects a comprehensive, systematic approach for assuring that data produced by the laboratory accurately reflects the outcome of the tests performed on field samples and has been produced in a bias free environment by ethical professionals. The policy includes a commitment to technical ethics, staff training in ethics and data integrity, an individual attestation to data integrity and procedures for evaluating data integrity. Senior management assumes the responsibility for assuring compliance with all technical ethics elements and operation of all data integrity procedures. The staff is responsible for compliance with the ethical code of conduct and for practicing data integrity procedures.

The Accutest Data Integrity Policy is as follows:

“Accutest Laboratories is committed to producing data that meets the data integrity requirements of the environmental regulatory community. This commitment is demonstrated through the application of a comprehensive data integrity program that includes ethics and data integrity training, data integrity evaluation procedures, staff participation and management oversight. Adherence to the specifications of the program assures that data provided to our clients is of the highest possible integrity and can be used for decision making processes with high confidence.”

Data Integrity Responsibilities

Management. Senior management retains oversight responsibility for the data integrity program and retains ultimate responsibility for execution of the data integrity program elements. Senior management is responsible for providing the resources required to conduct ethics training and operate data integrity evaluation procedures. They also include responsibility for creating an environment of trust among the staff and being the lead advocate for promoting the data integrity policy and the importance of technical ethics. The Quality Assurance Director is the designated ethics officer for the Company.

Staff. The staff is responsible for adhering to the company ethics policy as they perform their duties and responsibilities associated with sample analysis and reporting. By executing this responsibility, data produced by Accutest Laboratories retains its high integrity characteristics and withstands the rigors of all data integrity checks. The staff is also responsible for adhering to all laboratory requirements pertaining to manual data edits, data transcription and data traceability. These include the application of approved manual peak integration and documentation procedures. It also includes establishing traceability for all manual results calculations and data edits.

Ethics Statement. The Accutest ethics statement reflects the standards that are expected for businesses that provide environmental services to regulated entities and regulatory agencies on a commercial basis. The Ethics Policy is comprised of key elements that are essential to organizations that perform chemical analysis for a fee. As such, it focuses on elements related to personal, technical and business activities.

Accutest Laboratories provides analytical chemistry services on environmental matters to the regulated community. The data the company produces provides the foundation for determining the risk presented by a chemical pollutant to human health and the environment. The environmental industry is dependent upon the accurate portrayal of environmental chemistry data. This process is reliant upon a high level of scientific and personal ethics.

It is essential to the Company that each employee understands the ethical and quality standards required to work in this industry. Accordingly, Accutest has adopted a code of ethics, which each employee is expected to adhere to as follows:

- o Perform chemical analysis using accepted scientific practices and principles.
- o Perform tasks in an honest, principled and incorruptible manner inspiring peers & subordinates.
- o Maintain professional integrity as an individual.
- o Provide services in a confidential, honest, and forthright manner.
- o Produce results that are accurate and defensible.
- o Report data without any considerations of self-interest.
- o Comply with all pertinent laws and regulations associated with assigned tasks and responsibilities.

Data Integrity Procedures. Four key elements comprise the Accutest data integrity system. Procedures have been implemented for conducting data integrity training and for documenting that employees conform to the Accutest Data Integrity and Ethics policy.

The data integrity program consists of routine data integrity evaluation and documentation procedures to periodically monitor and document data integrity. These procedures are documented as SOPs. SOPs are approved and reviewed annually following the procedures employed for all Accutest SOPs. Documentation associated with data integrity evaluations is maintained on file and is available for review.

Data Integrity Training. Accutest employees receive technical ethics training during new employee orientation. Employees are also required to refresh their ethical conduct agreement annually, which verifies their understanding of ANC's ethics policy and their ethical responsibilities. A brochure summarizing the details of the Accutest Data Integrity Policy is distributed to all employees with the Ethical Conduct Agreement. The refreshed agreements are archived.

The training focuses on the reasons for technical ethic training, explains the impact of data fraud on human health and the environment, and illustrates the consequences of criminal fraud on businesses and individual careers. ANC's ethics policy and code of ethics are reviewed and explained for each new employee.

Training on data integrity procedures are conducted by individual departments for groups involved in data operations. These include procedures for manual chromatographic peak integration, traceability for manual calculations and data transcription.

Data Integrity Training Documentation. Records of all data integrity training are maintained in training folders. Attendance at all training sessions is documented and maintained in the training archive.

Accutest Data Integrity and Ethical Conduct Agreement. All employees are required to sign a Data Integrity and Ethical Conduct Agreement annually. This document is archived in training folder, which are retained for duration of employment.

The Data Integrity and Ethical Conduct Agreement is as follows:

- I. I understand the high ethical standards required of me with regard to the duties I perform and the data I report in connection with my employment at Accutest Laboratories-Northern California.*
- II. I have received formal instruction on the code of ethics that has been adapted by Accutest Laboratories-Northern California during my orientation and agree to comply with these requirements.*
- III. I have received formal instruction on the elements of Accutest Laboratories' Data Integrity Policy and have been informed of the following specific procedures:*
 - a. Formal procedures for the confidential reporting of data integrity issues are available, which can be used by any employee,*
 - b. A data integrity investigation is conducted when data issues are identified that may negatively impact data integrity.*
 - c. Routine data integrity monitoring is conducted on sample data, which may include an evaluation of the data I produce,*
- IV. I have read the brochure detailing Accutest Laboratories Data Integrity and Ethics Program as required.*
- V. I am aware that data fraud is a punishable crime that may include fines and/ or imprisonment upon conviction.*
- VI. I also agree to the following:*
 - a. I shall not intentionally report data values, which are not the actual values observed or measured.*
 - b. I shall not intentionally modify data values unless the modification can be technically justified through a measurable analytical process.*
 - c. I shall not intentionally report dates and times of data analysis that are not the true and actual times the data analysis was conducted.*
 - d. I shall not condone any accidental or intentional reporting of inauthentic data by other employees and immediately report it's occurrence to my superiors.*
 - e. I shall immediately report any accidental reporting of inauthentic data by myself to my superiors.*

Data Integrity Monitoring. Documented procedures are employed for performing data integrity monitoring. These include regular data review procedures by supervisory and management staff (Section 12.7), supervisory review and approval of manual integrations and periodic reviews of GALP audit trails from the LIMS and all computer controlled analysis.

Data Review. All data produced by the laboratory undergoes several levels of review, which includes two levels of management review. Detected data anomalies that appear to be related to data integrity issues are isolated for further investigation. The investigation is conducted following the procedures described in this section.

Manual Peak Integration Review and Approval. Routine data review procedures for all chromatographic processes includes a review of all manual chromatographic peak integrations. This review is performed by the supervisory staff and consists of a review of the machine integration compared to the manual integration. Manual integrations, which have been performed in accordance with ANC's manual peak integration procedures, are approved for further processing and release. Manual integrations which are not performed to ANC's specifications are set aside for corrective action, which may include analyst retraining or further investigation as necessary.

GALP Audit Trail Review. Good Automated Laboratory Practice (GALP) audits are comprehensive data package audits that include a review of raw data, process logbooks, processed data reports and GALP audit trails from individual instruments and LIMS. GALP audit trails, which record all electronic data activities, are available for the majority of computerized methodology and the laboratory information management system (LIMS). These audit trails are periodically reviewed to determine if interventions performed by technical staff constitute an appropriate action. The review is performed on a recently completed job and includes interviews with the staff that performed the analysis. Findings indicative of inappropriate interventions or data integrity issues are investigated to determine the cause and the extent of the anomaly.

Confidential Reporting Of Data Integrity Issues. Data integrity concerns may be raised by any individual to their supervisor. Employees with data integrity concerns should always discuss those concerns with their immediate supervisors as a first step unless, the employee is concerned with the confidentiality of disclosing data integrity issues or is uncomfortable discussing the issue with their immediate supervisors. The supervisor makes an initial assessment of the situation to determine if the concern is related to a data integrity violation. Those issues that appear to be violations are documented by the supervisor and referred to the Quality Assurance Officer for investigation.

Documented procedures for the confidential reporting of data integrity issues in the laboratory are part of the data integrity policy. These procedures assure that laboratory staff can privately discuss ethical issues or report items of ethical concern without fears of repercussions with senior staff.

Employees with data integrity concerns that they consider to be confidential are directed to the Corporate Human Resources Manager in Dayton, New Jersey. The HR Manager acts as a conduit to arrange a private discussion between the employee and the Corporate QA Director or a local QA Officer.

During the employee - QA discussion, the QA representative evaluates the situation presented by the employee to determine if the issue is a data integrity concern or a legitimate practice. If the practice is legitimate, the QA representative clarifies the process for the employee to assure understanding. If the situation appears to be a data integrity concern, the QA representative initiates a Data Integrity Investigation following the procedures specified in SOP_Data_Integrity_Investigations_20080215.doc.

Data Integrity Investigations. Follow-up investigations are conducted for all reported instances of ethical concern related to data integrity. Investigations are performed in a confidential manner by senior management according to a documented procedure. The outcome of the investigation is documented and reported to the company president who has the ultimate responsibility for determining the final course of action in the matter. Investigation documentation includes corrective action records, client notification information and disciplinary action outcomes, which is archived for a period of five years.

The investigations are conducted by the senior staff and supervisory personnel from the affected area. The investigations team includes the Laboratory Director and the Quality Assurance Director. Investigations are conducted in a confidential manner until it is completed and resolved.

The investigation includes a review of the primary information in question by the investigations team. The team performs a review of associated data and similar historical data to determine if patterns exist. Interviews are conducted with key staff to determine the reasons for the observed practices.

Following data compilation, the investigations team reviews all information to formulate a consensus conclusion. The investigation results are documented along with the recommended course of action.

Corrective Action, Client Notification & Discipline. Investigations that reveal systematic data integrity issues will be referred for an Incident Report. If the investigation indicates that an impact to data has occurred and the defective data has been released to clients, the client will be notified by the Laboratory Director.

In all cases of data integrity violations, some level of disciplinary action will be conducted on the responsible individual. The level of discipline will be consistent with the violation and may range from retraining and/or verbal reprimand to termination. A zero tolerance policy is in effect for unethical actions.

4.0 JOB DESCRIPTIONS OF KEY STAFF

- 4.1 **Requirement:** Descriptions of key positions within the organization are defined to ensure that clients and staff understand duties and the responsibilities of the management staff and the reporting relationships between positions.

President/Chief Executive Officer. Responsible for all laboratory operations and business activities. Establishes the company mission and objectives in response to business needs. Direct supervision of the Vice President of Operations, each laboratory director, client services, management information systems, quality assurance and health and safety.

Vice President, Operations (Corporate). Reports to the company president. Establishes laboratory operations strategy. Operational responsibility for Orlando, Florida, Marlborough, Massachusetts, Santa Clara, California and Houston, Texas laboratories. Assumes the responsibilities of the CEO in his absence.

Laboratory/Technical Director (Local). Reports to the Vice President of Operations. Establishes laboratory operations strategy. Direct supervision of organic chemistry, inorganic chemistry, and sample management. Minimum Qualifications: BS in Chemistry or other physical science and 10 years experience in Environmental Chemistry.

Vice President, Chief Information Officer (Corporate). Reports to the company president. Develops the IT software and hardware agenda. Provides system strategies to compliment company objectives. Maintains all software and hardware used for data handling.

Director, Quality Assurance (Corporate). Reports to the company president. Establishes the company quality agenda, develops quality procedures, provides assistance to operations on quality procedure implementation, coordinates all quality control activities, monitors the quality system, provides quality system feedback to management to be used for process improvement and oversees health and safety. Assumes the responsibilities of the CEO in the absence of the CEO and the Vice President Operations.

Manager, Volatile Organics . Reports to the laboratory director. Directs the operations of the volatile organics group, consisting of organics preparation and instrumental analysis. Establishes daily work schedule. Supervises method implementation, application, and data production. Responsible for following Quality System requirements. Maintains laboratory instrumentation in an operable condition. Minimum Qualifications: BS in Chemistry or other physical science and 5 years experience in Environmental Chemistry.

Manager, Semi-Volatile Organics . Reports to the laboratory director. Directs the operations of the Semi-volatile organics analysis and Organic extractions group. Establishes daily work schedule. Supervises method implementation, application, and data production. Responsible for following Quality System requirements. Maintains laboratory instrumentation in an operable condition. Minimum Qualifications: BS in Chemistry or other physical science and 5 years experience in Environmental Chemistry.

Inorganics Supervisor. Reports to the laboratory director. Supervises the operations of the inorganics group, consisting of wet chemistry and the metals laboratories. Maintains laboratory instrumentation in an operable condition. Establishes daily analysis schedule. Supervises method implementation, application, and data production. Supervises the analysis of samples using valid, documented methodology. Reviews data for compliance to quality and methodological requirements. Responsible for following Quality System requirements. Minimum Qualifications: BS in Chemistry or other physical science and 5 years experience in Environmental Chemistry.

Manager, Sample Management. Reports to the laboratory director. Develops, maintains and executes all procedures required for receipt of samples, verification of preservation, and chain of custody documentation. Responsible for maintaining and documenting storage, and courier services. Minimum Qualifications: BS degree and 5 years experience in Environmental Chemistry.

Principal Analysts. Reports to the department Supervisors. Maintains laboratory instrumentation in an operable condition, analyze samples on a daily basis. Implement methods, applications, and data production. Analyze samples using valid, documented methodology. Mentor junior analysts and reviews data for compliance to quality and methodological requirements. Responsible for following Quality System requirements. Minimum Qualifications: BS in Chemistry or other physical science and 1 years experience in Environmental Chemistry.

Health & Safety Officer. Reports to the Director of Quality Assurance. Responsible for developing company safety program and chemical hygiene plan. Reviews and updates these plans annually. Responsible for employee training on relevant health and safety topics. Documents employee training. Manages laboratory waste management program.

Quality Assurance Officer (Local). Reports to the Director of Quality Assurance. Performs quality control data review for trend monitoring purposes. Conducts internal audits and prepares reports for management review. Oversees proficiency testing program. Process quality control data for statistical purposes.

4.2 Employee Screening, Orientation, and Training

All potential laboratory employees are screened and interviewed by human resources and technical staff prior to their hire. The pre-screen process includes a review of their qualifications including education, training and work experience to verify that they have adequate skills to perform the tasks of the job.

Newly hired employees receive orientation training beginning the first day of employment by the Company. Orientation training consists of initial health and safety training including general laboratory safety, personal protection and building evacuation. Orientation also includes quality assurance program training, data integrity training, and an overview of the Company's goals, objectives, mission, and vision.

All technical staff receives training to develop and demonstrate proficiency for the methods they perform. New analysts work under supervision until the supervisory staff is satisfied that a thorough understanding of the method is apparent and method proficiency has been demonstrated, through a precision and accuracy study that has been documented, reviewed and approved by the QA Officer. Data from the study is compared to method acceptance limits. If the data is unacceptable, additional training is required. The analyst may also demonstrate proficiency by producing acceptable data through the analysis of an independently prepared proficiency sample.

Individual proficiency is demonstrated annually for each method performed. ODOC Data from initial and continuing proficiency demonstrations are archived in the individual's training folder.

4.3 Training Documentation. The human resources department prepares a training file for every new employee. This administrative information related to qualifications, experience, external training courses, and education are placed into the file. Verification documentation for orientation, health & safety, quality assurance, and ethics training is also included in the file.

Additional technical training documentation is filed locally. This includes documentation of SOP understanding, data for initial and continuing demonstrations of proficiency, performance evaluation study data and notes and attendance lists from group training sessions.

The Quality Assurance Department maintains the employee training directory. This directory is a comprehensive inventory of training documentation for each individual employee. These files enable the supervisors to obtain current status information on technical training data for individual employees.

5.0 SIGNATORY APPROVALS

Requirement: Procedures have been developed for establishing the traceability of data and documents. The procedure consists of a signature hierarchy, indicating levels of authorization for signature approvals of data and information within the organization. Signature authority is granted for approval of specific actions based on positional hierarchy within the organization and knowledge of the operation that requires signature approval. A log of signatures and initials of all employees is maintained by the HR Staff for cross-referencing purposes.

5.1 Signature Hierarchy.

President/Chief Executive Officer. Authorization for contracts and binding agreements with outside parties. Approval of final reports, quality assurance policy, SOPs, project specific QAPs, data review and approval in lieu of technical managers. Note: Contract signature authority resides with Company officers only, which include the President/CEO, Chief Financial Officer and Vice President Administration.

Vice President, Operations/Laboratory Director. Approval of final reports and quality assurance policy in the absence of the President. Approval of SOPs, project specific QAPs, data review and approval in lieu of technical managers. Establishes and implements technical policy.

Director, Quality Assurance. Approval of final reports and quality assurance policy in the absence of the President. Approval of SOPs, project specific QAPs, data review and approval in lieu of technical managers.

Director, Client Services. QAP and sampling and analysis plan approval. Project specific contracts, pricing, and price modification agreements. Approval and acceptance of incoming work, Client services policy.

Managers, Technical Departments. Methodology and department specific QAPs. Data review and approval, department specific supplies purchase. Technical approval of SOPs.

Manager, Sample Management. Initiation of laboratory sample custody and acceptance of all samples. Approval of department policies and procedures. Department specific supplies purchase.

Manager, Health & Safety. Approval of health and safety policy in the absence of the President and QA Director. Approval of health and safety SOPs. Waste manifesting and approval.

Supervisors, Technical Departments. Data review approval, purchasing of expendable supplies.

- 5.2 **Signature Requirements.** All laboratory activities related to sample custody and generation or release of data must be approved using either initials, signatures or electronic, password protected procedures. The individual, who applies his signature initial or password to an activity or document, is authorized to do so within the limits assigned to them by their supervisor. All written signatures and initials must be applied in a readable format that can be cross-referenced to the signatures and initials log if necessary.
- 5.3 **Signature and Initials Log.** The Laboratory maintains a signature and initials log. New employee signatures and initials are appended to the log on the first day of employment. Signature of individuals no longer employed by the company are retained, but annotated with their date of termination.

6.0 DOCUMENTATION & DOCUMENT CONTROL

Requirement. Document control policies have been established which specify that any document used as an information source or for recording analytical or quality control information must be managed using defined document control procedures. Accordingly, policies and procedures required for the control, protection, and storage of any information related to the production of analytical data and the operation of the quality system to assure its integrity and traceability have been established and implemented in the laboratory. The system contains sufficient controls for managing, archiving and reconstructing all process steps which contributed to the generation of an analytical test result. Using this system, an audit trail for reported data can be produced, establishing complete traceability for the result.

- 6.1 **Administrative Records.** Administrative (non-analytical) records are managed by the quality assurance department. These records consist of electronic documents which are retained in a limited access electronic directory or paper documents, which are released to the technical staff upon specific request.

Form Generation, Modification & Control. The quality assurance group approves and manages all forms used as either stand-alone documents or in logbooks to ensure their traceability. Forms are generated as computer files only and are maintained in a limited access master directory. The QA staff also manages and approves modifications to existing forms. Obsolete editions of modified forms are retained for seven years.

New forms must include the name Accutest Laboratories and appropriate spaces for signatures of approval and dates. Further design specifications are the responsibility of the originating department.

The technical staff is required to complete all forms to the maximum extent possible. If information for a specific item is unavailable, the analyst is required to "Z" the information block. The staff is also required to "Z" the uncompleted portions of a logbook or logbook form if the day's analysis does not fill the entire page of the form.

Logbook Control. All laboratory logbooks are controlled documents that are comprised of approved forms used to document specific processes. New logs are numbered and issued to a specific individual who is assigned responsibility for the log. Old logs are returned to QA for entry into the document archive system where they are retained for seven (7) years. Laboratory staff may hold a maximum of two consecutively dated logbooks of the same type in the laboratory including the most recently issued book to simplify review of recently completed analysis.

Controlled Documents. Key laboratory documents that are distributed internally and externally are numbered and dated for tracking purposes. Individuals receiving documents, who must be assured they have the most recent, receive a file server location where the most current revisions reside. Control is maintained through a document - revision numbering and dating procedure. Key documents are also distributed as uncontrolled documents if the recipient does not require updated copies when changes occur.

Quality Systems Manual (QSM). All QSMs are titled, revision indicated, and dated prior to distribution. Electronic versions are distributed as read only files that are password protected. **The online version is always the most current.**

Standard Operating Procedures (SOPs). SOPs are maintained by title, revision, and date. One copy of the SOP is placed into each the department. SOPs are reviewed annually.

The original, signed copy of the SOP is maintained in the master SOP binder by the QA staff. The QA staff collects outdated versions of SOPs as they are replaced and archived for a period of seven (7) years in the QA archives. Electronic versions of outdated SOPs are moved from the active SOP directory to the inactive directory identified by year.

- 6.2 **Technical Records.** All records related to the analysis of samples and the production of an analytical result are archived in secure document storage or on electronic media and contain sufficient detail to produce an audit trail which re-creates the analytical result. These records include information related to the original client request, bottle order, sample login and custody, storage, sample preparation, analysis, data review and data reporting.

Each department involved in this process maintains controlled documents which enable them to maintain records of critical information relevant to their department's process.

- 6.3 **Quality Control Support Data & Records.** All information and data related to the quality system is stored in a restricted access directory on the network server. Information on this directory is backed-up daily. Users of the quality assurance information and data have "read-only" access to the files contained in the directory. The QA staff and the laboratory director have write capability in this directory.

This directory contains all current and archived quality system manuals, SOPs, control limits, MDL studies, precision and accuracy data, official forms, internal audit reports, proficiency test scores and metrics calibration information.

- 6.4 **Analytical Records.** All data related to the analysis of field samples are retained as either paper or electronic records that can be retrieved to compile a traceable audit trail for any reported result. All information is linked to the client job and sample number, which serves as a reference for all sample related information tracking.

Critical times in the life of the sample from collection through analysis to disposal are documented. This includes date and time of collection, receipt by the laboratory, preparation times and dates, analysis times and dates and data reporting information. Analysis times are calculated in hours for methods where holding time is specified in hours (≤ 72 hours).

Sample preparation information is recorded in a separate controlled logbook. It includes sample identification numbers, types of analysis, preparation and cleanup methods, sample weights and volumes, reagent lot numbers and volumes and any other information pertinent to the preparation procedure.

Information related to the identification of the instrument used for analysis is permanently attached to the electronic record. The record includes an electronic data file that indicates all instrument conditions employed for the analysis, including the type of analysis conducted. The analyst's identification is electronically attached to the record. The instrument tuning and calibration data is electronically linked to the sample or linked through paper logs which were used in the documentation of the analysis. Quality control and performance criteria are permanently linked to the paper archive or electronic file.

Paper records for the identity, receipt, preparation and evaluation of all standards and reagents used in the analysis are documented in prepared records and maintained in controlled documents or files. Lot number information linking these materials to the analysis performed is recorded in the logbooks associated with the samples in which they were used.

Manual calculations or peak integrations that were performed during the data review are retained as paper or scanned documents and included as part of the electronic archive. Signatures for data review are retained on paper or as scanned versions of the paper record for the permanent electronic file.

- 6.5 **Confidential Business Information (CBI).** Operational documents including SOPs, Quality Manuals, personnel information, internal operations statistics, and laboratory audit reports are considered confidential business information. Strict controls are placed on the release of this information to outside parties.

Release of CBI to outside parties or organizations may be authorized upon execution of a confidentiality agreement between Accutest and the receiving organization or individual. CBI information release is authorized for third party auditors and commercial clients in electronic mode as secured Adobe Acrobat .PDF format only.

- 6.6 **Software Change Documentation & Control.** Changes to software are documented as text within the code of the program undergoing change. Documentation includes a description of the change, reason for change and the date the change was placed into effect. Documentation indicating the adequacy of the change is prepared following the evaluation by the user who requested the change.

- 6.7 **Report and Data Archiving.** Accutest Laboratories produces digital files of all raw and processed data which is maintained for a minimum period of seven (7) years. The archived files consist of all raw data files and source documents associated with the analysis of field samples and proficiency test samples. Data files and source documents associated with method calibration and project and method quality control are also archived. After seven years, the files are discarded unless contractual arrangements exist which dictate different requirements. Client or regulatory agency specific data retention practices are employed for several government organizations such as the Department of Defense and the Massachusetts Department of Environmental Protection that require a retention period of ten (10) years. Data archiving may also be extended up to ten (10) years for specific commercial clients in response to contractual requirements.

Complete date and time stamped PDF reports are generated from the laboratory information management system (LIMS) using the source documents archived on the document server. These source documents are maintained on a document server and archived to primary and clone tapes. The primary tapes remain on premises while the clone tapes are taken to a secure offsite location for permanent storage. Both the primary and clone tapes remain in storage for the remainder of the archive period.

- 6.8 **Training:** The company maintains a training record for all employees that documents that they have received instruction on administrative and technical tasks that are required for the job they perform. Training records for individuals employed by the company are retained for a period of six months following their termination of employment.

Training File Origination. The Human Resources Group (HR) initiates training files. The QA staff, through the Assistant Quality Assurance officer, retains the responsibility for the maintenance and tracking of all training related documentation in the file. The file is begun on the first day of employment. Information required for the file includes a copy of the individual's most current resume, detailing work experience and a copy of any college diplomas and transcript(s). Information added on the first day includes documentation of health and safety training, quality assurance training and a signed data integrity training and ethical conduct agreement.

Training documentation, training requirements, analyst proficiency information and other training related support documentation is archived locally.

- 6.9 **Technical Training:** The supervisor of each new employee is responsible for developing a training plan for each new employee. The supervisor evaluates the employees training progress at regular frequencies. Supporting documentation, including demonstration of capability and precision and accuracy studies, which demonstrate an analyst's proficiency for a specific test, are added to the training file as completed. Employees and supervisors verify documentation of understanding for all assigned standard operating procedure. Certificates or diplomas for any off-site training are also added to the file.

7.0 REFERENCE STANDARD TRACEABILITY

Requirement: Documented procedures, which establish traceability between any measured value and a national reference standard, are established by the laboratory as required. All metric measurements are traceable to NIST reference weights or thermometers that are calibrated on a regular schedule. All chemicals used for calibration of a quantitative process are traceable to an NIST reference that is documented by the vendor using a certificate of traceability. The laboratory maintains a documentation system that establishes the traceability links. The procedures for verifying and documenting traceability are documented in standard operating procedures.

7.1 **Traceability of Metric Measurements - Thermometers.** ANC uses NIST thermometers to calibrate commercially purchased thermometers prior to their use in the laboratory. If necessary, thermometers are assigned correction factors that are determined during their calibration using an NIST thermometer as the standard. The correction factor is documented on a tag attached to the thermometer. The correction factor is applied to temperature measurements before recording the measurement in the temperature log. Calibration of each thermometer is verified and documented on a regular schedule. The NIST thermometer is checked for accuracy by a qualified vendor every five (5) years following the specifications for NIST thermometer calibration verification detailed in the United States Environmental Protection Agency's "Manual for the Certification of Laboratories Analyzing Drinking Water", Fifth Edition, January 2005.

7.2 **Traceability of Metric Measurements – Calibration Weights.** Accutest uses calibrated weights, which are traceable to NIST standard weights to calibrate all balances used in the laboratory. Balances are calibrated to specific tolerances within the intended use range of the balance. Calibration checks are required on each day of use. If the tolerance criteria are not achieved, corrective action specified in the balance calibration SOP is applied before the balance can be used for laboratory measurements. Recalibration of all calibration weights is conducted and documented on a biannual basis.

7.3 **Traceability of Chemical Standards.** All chemicals, with the exception of bulk dry chemicals and acids, purchased as reference standards for use in method calibration must establish traceability to NIST referenced material through a traceability certificate. Process links are established that enable a calibration standard solution to be traced to its NIST reference certificate.

Chemical standards used for analysis must meet the purity specifications of the method. These specifications must be stated in the reagents section of the method SOP.

7.4 **Assignment of Reagent and Standard Expiration Dates.** Expiration date information for all purchased standards, prepared standard solutions and selected reagents is provided to Accutest by the vendor as a condition of purchase. Neat materials and inorganic reagents are not required to be purchased with expiration dates. Prepared solutions are labeled with the

expiration date provided by the manufacturer. In-house prepared solutions are assigned expiration dates that are consistent with the method that employs their use unless documented experience indicates that an alternate date can be applied. If alternate expiration dates are employed, their use is documented in the method SOP. Expiration dates for prepared inorganic reagents, which have not exhibited instability, are established at two years from the date of preparation for tracking purposes.

The earliest expiration date has been established as the limiting date for assigning expiration dates to prepared solutions. The assignments of expiration dates that are later than the expiration date of any derivative solution or material are prohibited.

- 7.5 **Documentation of Traceability.** Traceability information is documented in individual logbooks designated for specific measurement processes. The quality assurance group maintains calibration documentation for metric references in separate logbooks.

Balance calibration verification is documented in logbooks that are assigned to each balance. The individual conducting the calibration is required to initial and date all calibration activities. Any defects that occur during calibration are also documented along with the corrective action applied and a demonstration of return to control. Annual service reports and certificates are retained on the file server by the QA staff.

Temperature control is documented in logbooks assigned to the equipment being monitored. A calibrated thermometer is assigned to each individual item. Record date and initials of the individual conducting the measurement on a daily or as used basis. Corrective action, if required, is also documented including the demonstration of return to control.

Initial traceability of chemical standards is documented via a vendor-supplied certificate (not available for bulk dry chemicals and acids) that includes lot number, expiration date and certified concentration information. Solutions prepared using the vendor supplied chemical standards are documented in logbooks assigned to specific analytical processes. The documentation includes links to the vendor's lot number, an internal lot number, dates of preparation, expiration date, and the preparer's initials.

Accutest employs commercially prepared standard solutions whose traceability can be demonstrated through a vendor supplied certificate of analysis that includes an experimental verification of the standard's true concentration. The test value for the verification analysis must agree within 1% of the vendor's true value before it can be employed for calibration purposes. If the test value differs from the nominal value by more than 1%, then the test value is used as the true value in laboratory calibrations and calculations. Purchased standards which do not have a certificate of analysis cannot be used for calibration or calibration verification purposes and are rejected or returned to the vendor.

Supervisors conduct regular reviews of logbooks, which are verified using a signature and date.

8.0 TEST PROCEDURES, METHOD REFERENCES, AND REGULATORY PROGRAMS

Requirements: The laboratory employs client specified or regulatory agency approved methods for the analysis of environmental samples. A list of active methods is maintained, which specifies the type of analyses performed and cross-references the methods to applicable environmental regulations. Routine procedures used by the laboratory for the execution of a method are documented in standard operating procedures. Method performance and sensitivity are demonstrated annually where required. Defined procedures for the use of method sensitivity limits for data reporting purposes are established by the Director of Quality Assurance and used consistently for all data reporting purposes.

- 8.1 **Method Selection & Application.** Accutest employs methods for environmental sample analysis that are consistent with the client's application, which are appropriate and applicable to the project objectives. Accutest informs the client if the method proposed is inappropriate or outdated and suggests alternative approaches.

Accutest employs documented, validated regulatory methods in the absence of a client specification and informs the client of the method selected. These methods are available to the client and other parties as determined by the client. Documented and validated in-house methods may be applied if they are appropriate to the project. The client is informed of the method selection.

- 8.2 **Standard Operating Procedures.** Standard operating procedures (SOP) are prepared for routine methods executed by the laboratory, processes related to laboratory operations and sample or data handling. All SOPs are formatted to meet the specifications established by the National Environmental Laboratory Accreditation Conference, which are detailed in Chapter Five – Quality Systems of the established Standards. The procedures describe the process steps in sufficient detail to enable an individual, who is unfamiliar with the procedure to execute it successfully.

SOPs are evaluated annually and edited if necessary. Reviewed SOPs that do not require modification include an evaluation summary form indicating that an evaluation was conducted and modifications were not needed. SOPs can be edited on a more frequent basis if changes are required for any reason. These may include a change to the methodology, elimination of systematic errors that dictate a need for process changes or modifications to incorporate a new version of the method promulgated by the originating regulatory agency. Procedural modifications are indicated using a revision number. SOPs are available for client review at the Accutest facility upon request.

- 8.3 **Method Validation.** Standard methods from regulatory sources are primarily used for all analysis. Standard methods do not require validation by the laboratory. Non-standard, in-house methods are validated prior to use. Validation is also performed for standard methods applied outside their intended scope of use. Validation is dependent upon the method application and may include analysis of quality control samples to develop precision and accuracy information for the intended use. A final method validation report is generated, which includes all data in the validation study. A statement of adequacy and/or equivalency is included in the report. A copy of the report is archived in the quality assurance directory of the company server.

Non-standard methods are validated prior to use. This includes the validation of modified standard methods to demonstrate comparability with existing methods. Demonstrations and validations are performed and documented prior to incorporating technological enhancements and non standard methods into existing laboratory methods used for general applications. The demonstration includes method specific requirements for assuring that significant performance differences do not occur when the enhancement is incorporated into the method. Validation is dependent upon method application and may include the analysis of quality control samples to develop precision and accuracy information for intended use.

The study procedures and specifications for demonstrating validation include comparable method sensitivity, calibration response, method precision; method accuracy and field sample consistency for several classes of analytical methods are detailed in this document. These procedures and specifications may vary depending upon the method and the modification.

- 8.4 **Estimated Uncertainty.** A statement of the estimated uncertainty of an analytical measurement accompanies the test result when required. Estimated uncertainty is derived from the performance limits established for spiked samples of similar matrices. The degree of uncertainty is derived from the negative or positive bias for spiked samples accompanying a specific parameter. When the uncertainty estimate is applied to a measured value, the possible quantitative range for that specific parameter at that measured concentration is defined. Well recognized regulatory methods that specify values for the major sources of uncertainty and specify the data reporting format do not require a further estimate of uncertainty.
- 8.5 **Demonstration of Capability.** Confirmation testing is conducted to demonstrate that the laboratory is capable of performing the method before its application to the analysis of environmental samples. The results of the demonstration tests are compared to the quality control specifications of the method to determine if the performance is acceptable.

Capability demonstrations are conducted initially for each method on every instrument and annually on a method specific basis thereafter. Acceptable demonstrations are documented for individual training files and retained by the QA staff. New analytes, which are added to the list of analytes for an accredited method, are evaluated for applicability through a demonstration of capability similar to those performed for accredited analytes.

- 8.6 **Method Detection Limit Determination.** Annual method detection limit (MDL) studies are performed as appropriate for routine methods used in the laboratory. MDL studies are also performed when there is a change to the method that affects how the method is performed or when an instrumentation change that impacts sensitivity occurs. The procedure used for determining MDLs is described in 40 CFR, Part 136, Appendix B. Studies are performed for each method on water, soil and air matrices for every instrument that is used to perform the method. MDLs are established at the instrument level. The highest MDL of the pooled instrument data is used to establish a laboratory MDL. The quality assurance staff manages the annual MDL determination process and is responsible for retaining MDL data on file. Approved MDLs are appended to the LIMS and used for data reporting purposes.
- 8.7 **Instrument Detection Limit Determination.** Instrument detection limits (IDLs) are determined for all inductively coupled argon plasma emission spectrophotometers and mass spectrometers. The IDL is determined for the wavelength (emission) of each element and the ion (mass spectrometry) of each element used for sample analysis. The IDL data is used to estimate instrument sensitivity in the absence of the sample matrix. IDL determinations are conducted at the frequency specified in the appropriate SOPs for ICP analysis.
- 8.8 **Method Reporting Limit.** The method reporting limit for organic methods is determined by the concentration of the lowest calibration standard in the calibration curve. This value is adjusted based on several sample preparation factors including sample volume, digestion, distillation or dilution. The low calibration standard is selected by department managers as the lowest concentration standard that can be used for calibration while continuing to meet the calibration linearity criteria of the method being used. The validity of the method reporting limits are confirmed through the analysis of a spiked quality control sample at the method reporting limit concentration. By definition, detected analytes at concentrations below the low calibration standard cannot be accurately quantitated and are qualified as estimated values.
- The reporting limits for inorganic methods is defined as the concentration which is greater than or equal to the MDL where method quality control criteria has been achieved. The reporting limit for general chemistry methods employing multiple point calibrations must be greater than or equal to the concentration of the lowest standard of the calibration range.
- 8.9 **Reporting of Quantitative Data.** Analytical data for all methods is reported without qualification to the reporting limit established for each method. Data, for organic methods may be reported to the established method detection limit depending upon the client's requirements provided that all qualitative identification criteria for the detected parameter have been satisfied. All parameters reported at concentrations between the reporting limit and the method detection limit is qualified as estimated.

Data for inorganic methods are reported to the established method reporting limits. Inorganic data for specific methods may also be reported to the established method detection limit at client request. However, this data is always qualified as estimated.

Measured concentrations of detected analytes that exceed the upper limit of the calibration range are either diluted into the range and reanalyzed or qualified as an estimated value. The only exception to this applies to ICP analysis, which can be reported to the upper limit of the experimentally determined linear range without qualification.

- 8.10 **Precision and Accuracy Studies.** Annual precision and accuracy (P&A) studies, which demonstrate the laboratories ability to generate acceptable data, are performed for all routine methods used in the laboratory. The procedure used for generating organic P&A data is referenced in the majority of the regulatory methodology in use. The procedure requires quadruplicate analysis of a sample spiked with target analytes at a concentration in the working range of the method. This data may be compiled from a series of existing blank spikes or laboratory control samples. Accuracy (percent recovery) of the replicate analysis is averaged and compared to established method performance limits. Values within method limits indicate an acceptable performance demonstration. Precision and accuracy data is also used to annually demonstrate analytical capability for individual analysts. Annual demonstration of capability data is archived in individual training files.

- 8.11 **Method Sources & References.** The Quality Assurance Staff maintains a list of active methods used for the analysis of samples. This list includes valid method references from sources such as USEPA, ASTM or Standard Methods designations and the current version and version date.

Updated versions of approved reference methodology are placed into use as changes occur. The Quality Assurance Director informs operations management of changes in method versions as they occur. The operations management staff selects an implementation date. The operations staff is responsible for completing all method use requirements prior to the implementation date. This includes modification of SOPs, completion of MDL and precision and accuracy studies and staff training. Documentation of these activities is provided to the QA staff who retains this information on file. The updated method is placed into service on the implementation date and the old version is de-activated.

Multiple versions of selected methods may remain in use to satisfy client specific needs. In these situations, the default method version becomes the most recent version. Client specific needs are communicated to the laboratory staff using method specific analytical method codes, which clearly depict the version to be used. The old method version is maintained as an active method until the specified client no longer requires the use of the older version.

Accutest will not use methodology that represents significant departures from the reference method unless specifically directed by the client. If clients direct the laboratory to use a method modification that represents a significant departure from the reference method, the request will be documented in the client file.

- 8.12 **Analytical Capabilities.** Appendix III provides a detailed listing of the methodology employed for the analysis of test samples.

9.0 SAMPLING, SAMPLE MANAGEMENT, LOGIN, CUSTODY, STORAGE AND DISPOSAL

Requirement: The laboratory must employ a system which ensures that client samples are adequately evaluated, acknowledged, and secured upon delivery to the laboratory. The system also assures that product chain of custody is maintained and that sample receipt conditions and preservation status are documented and communicated to the client and internal staff. The login procedure assigns, documents, and maps the specifications for the analysis of each unique sample to assure that the requested analysis is performed on the correct sample and enables the sample to be tracked throughout the laboratory analytical cycle. The system includes procedures for reconciling defects in sample condition or client provided data, which are identified at sample arrival. The system specifies the procedures for proper sample storage, transfer to the laboratory, and disposal after analysis. The system is also documented in standard operating procedures.

- 9.1 **Order Receipt and Entry:** New orders are initiated and processed by the client services group. The new order procedure includes mechanisms for providing bottles to clients, which meet the size, cleanliness, and preservation specifications for the analysis to be performed.

For new orders, the project manager prepares a bottle request form, which is submitted to sample management. This form provides critical project details to the sample management staff, which are used to prepare and assemble the sample bottles for shipment to the client prior to sampling.

The bottle order is assembled using bottles that meet USEPA specifications for contaminant free sample containers. Accutest-Northern California uses commercially supplied pre-cleaned bottles.

Reagent water for trip and field blanks is poured into appropriately labeled containers. Bottles may be packed into ice chests with blank chain of custody forms and the original bottle order form or bottle shipped alone. Completed bottle orders are delivered to clients using Accutest couriers or commercial carriers for use in field sample collection.

- 9.2 **Sampling:** Accutest-Northern California does not have a sampling staff.

- 9.3 **Sample Receipt and Custody:** Samples are delivered to the laboratory using a variety of mechanisms including Accutest couriers, commercial shippers, and client self-delivery. Documented procedures are followed for arriving samples to assure that custody and integrity are maintained and handling/ preservation requirements are documented and maintained.

Sample custody documentation is initiated when the individual collecting the sample collects field samples. Custody documentation includes all information necessary to provide an unambiguous record of sample collection, sample identification, and sample collection

chronology. Initial custody documentation employs either Accutest or client generated custody forms.

Accutest generates a chain of custody in situations where the individuals who collected the sample did not generate custody documentation in the field.

Accutest defines sample custody as follows:

- ∴ The sample is in the actual custody or possession of the assigned responsible person,
- ∴ The sample is in a secure area.

The Accutest facility is defined as a secure facility. Perimeter security has been established, which limits access to authorized individuals only. The sample management team is located in the vicinity of all sample storage during work hours. Visitors enter the facility through the building lobby and must register with the receptionist prior to entering controlled areas. While in the facility, visitors must be accompanied by their hosts at all times. After hours, building access is controlled using a computerized alarm system. This system limits building access to individuals with a pre-assigned authorization status. After hours visitors are not authorized to be in the building. Clients delivering samples after hours must make advanced arrangements through client services and sample management to assure that staff is available to take delivery and maintain custody.

Upon arrival at Accutest, the sample custodian reviews the chain of custody for the samples received to verify that the information on the form corresponds with the samples delivered. This includes verification that all listed samples are present and properly labeled, checks to verify that samples were transported and received at the required temperature, verification that the sample was received in proper containers, verification that sufficient volume is available to conduct the requested analysis, and a check of individual sample containers to verify test specific preservation requirements including the absence of headspace for volatile compound analysis.

Sample conditions and other observations are documented on the chain of custody by the sample custodian prior to completing acceptance of custody and in the LIMS. The sample custodian accepts sample custody upon verification that the custody document is correct. Discrepancies or non-compliant situations are documented and communicated to the Accutest project manager, who contacts the client for resolution. The resolution is documented in the report archive directory on the file server and communicated to sample management for execution.

During initial login, each sample is assigned a unique number and is labeled with that number.

- 9.4 **Laboratory Preservation of Improperly Preserved Field Samples.** Accutest will attempt to preserve field samples that were received without proper preservation to the extent that it is feasible and supported by the methods in use. Laboratory preservation of improperly preserved or handled field samples is routinely performed for metals samples. Special handling procedures may also be applied to improperly preserved volatile organics.

Aqueous metals samples that were not nitric acid preserved to pH 2 in the field are laboratory preserved and held for twenty-four (24) hours to equilibrate prior to analysis (per Federal Register). Aqueous metals samples requiring field filtration may be filtered in the laboratory within seventy-two (72) hours of receipt provided that the sample has not been acid preserved.

Unpreserved volatile organics samples may be analyzed within seven (7) days to minimize degradation of volatile organics if the laboratory is notified in advance of the failure to preserve upon collection. Laboratory preservation of unpreserved aqueous samples is not possible. A pH check of volatile organic samples prior to analysis will compromise the sample by allowing volatile organics to escape during the check. If the laboratory is not notified of the failure to field preserve an aqueous volatile organic sample, the defect will not be identified until sample analysis has been completed and the data is qualified accordingly.

- 9.5 **Sample Tracking Via Status Change.** An automated, electronic LIMS procedure records sample exchange transactions between departments and changes in analytical status. This system tracks all preparation, analytical, and data reporting procedures to which a sample is subjected while in the possession of the laboratory.

Sample tracking is initiated at login where all chronological information related to sample collection dates and holding times are entered into the LIMS. This information is entered on an individual sample basis.

- 9.6 **Sample Acceptance Policy.** Incoming samples must satisfy ANC's sample acceptance criteria before being logged into the system. Sample acceptance is based on the premise that clients have exercised proper protocols for sample collection. This includes complete documentation, sufficient volume, proper chemical preservation, temperature preservation, sample container sealing and labeling, and appropriate shipping container packing.

The sample management staff will make every attempt to preserve improperly preserved samples upon arrival. However, if preservation is not possible, the samples may be refused unless the client authorizes analysis. No samples will be accepted if holding times have been exceeded or will be exceeded before analysis can take place unless the client authorizes analysis.

Sample acceptance criteria include proper custody and sample labeling documentation. Proper custody documentation includes an entry for all physical samples delivered to the laboratory with an identification code that matches the sample bottle and a date and signature of the individual who collected the sample and delivered them to the laboratory.

Accutest-Northern California reserves the right to refuse any sample which in its sole and absolute discretion and judgment is hazardous, toxic and poses or may pose a health, safety or environmental risk during handling or processing. The company will not accept samples for analysis using methodology that is not performed by the laboratory or for methods that lab does not hold valid accreditations unless arrangements have been made to have the analysis conducted by a qualified subcontractor.

- 9.7 **Assignment of Unique Sample Identification Codes.** Unique identification codes are assigned to each sample to assure traceability and unambiguously identify the tests to be performed in the laboratory.

The sample identification coding process begins with the assignment of a unique alphanumeric job number. A job is defined as a group of samples received on the same day, from a specific client pertaining to a specific project. A job may consist of groups of samples sampled over a multi-day period. The first character of the job number is an alpha-character that identifies the laboratory facility. Accutest Northern California has job numbers that start with "C". The next characters are numeric and sequence by one number with each new job.

Unique sample numbers are assigned to each bottle collected as a discrete entity from a designated sample point. This number begins with the job number and incorporates a second series of numbers beginning at one and continuing chronologically for each point of collection.

Alpha suffixes may be added to the sample number to identify special designations such as subcontracted tests, in-house QC checks, or re-logs.

- 9.8 **Subcontracted Analysis.** Subcontract laboratories are employed to perform analysis not performed by Accutest. The quality assurance staff evaluates subcontract laboratories via CDPH-ELP and NELAP certification to assure their quality processes meet the standards of the environmental laboratory industry prior to engagement. Throughout the subcontract process, Accutest follows established procedures to assure that sample custody is maintained and the data produced by the subcontractor meets established quality criteria.

Subcontracting Procedure. Subcontracting procedures are initiated through several mechanisms, which originate with sample management. Samples for analysis by a subcontractor are logged into the Accutest system using regular login procedures. If subcontract parameters are part of the project or sample management has received subcontracting instructions for a specific project, a copy of the chain of custody is given to the appropriate project manager with the subcontracted parameters highlighted. This procedure triggers the subcontract process at the project management level. The project manager contacts an approved subcontractor that carries accreditation in the venue of the project location to place the subcontract order. A subcontract chain of custody is simultaneously prepared in electronic format and filed with the original chain of custody. The subcontract chain of custody is forwarded along with the samples.

Sample management signs the subcontract chain of custody and ships the sample(s) to the subcontractor. The subcontract CoC is filed with the original CoC and the request for subcontract.

Clients are verbally notified of the need to subcontract analysis as soon as the need is identified by the client services staff. This may occur during the initial project setup or at the time of login if the project setup had not been initiated through the client services staff. Copies of the subcontract CoC and the original CoC, which are electronically distributed to clients, this constitutes documented client notification of the laboratories' intent to subcontract analyses.

Subcontractor data packages are reviewed by the Reporting Staff to assess completeness. If completeness defects are detected, the subcontractor is asked to immediately upgrade the data package.

Subcontract data is wholly incorporated into the final report by Adobe PDF Acrobat.

Subcontract Laboratory Evaluation. The subcontract laboratory must provide ANC with proof of a valid certification to perform the requested analysis for the venue where they were collected, and a copy of the laboratory's Quality Systems Manual. If possible, the QA staff may conduct a site visit to the laboratory to inspect the quality system. Qualification of a subcontract laboratory is bypassed if the primary client directs Accutest-Northern California to employ a specific subcontractor. All subcontracted items for testing covered under DoD will only be submitted to a DoD-ELAP laboratories.

- 9.9 **Sample Storage.** Following sample transfer to the sample custodian, samples are assigned to various refrigerated storage areas depending upon the test to be performed and the matrix of the samples. Samples remain in storage until the laboratory technician removes them for analysis.

The Accutest facility is defined as a secure facility. Perimeter security has been established, which limits access to authorized individuals only. Visitors enter the facility through the building lobby and must register with the receptionist prior to entering controlled areas. While in the facility, visitors must be accompanied by their hosts at all times. After hours, building access is controlled using an alarm system. After hours visitors are not authorized to be in the building. Clients delivering samples after hours must make advanced arrangements through client services and sample management to assure that staff is available to take delivery and maintain custody.

Samples for volatile organics analysis are placed in specifically designated refrigerators. These samples are segregated according to matrix to limit opportunities for cross contamination to occur.

Organics staff is authorized to retrieve samples from these storage areas for analysis. When analysis is complete, the samples are placed back into storage.

- 9.10 **Sample Login.** Following sample custody transfer to the laboratory, the documentation that describes the clients analytical requirements are delivered to the sample login group for coding and entry to the Laboratory Information Management System (LIMS). This process translates all information related to collection time, turnaround time, sample analysis, and deliverables into a code which enables client requirements to be electronically distributed to the various departments within the laboratory for scheduling and execution. The technical staff is alerted to client or project specific requirements through the use of commenting in the LIMS.
- 9.11 **Sample Retrieval for Analysis.** Retrieval priorities are established by the requesting department and submitted to the sample custodian when multiple requests are submitted. After sample analysis has been completed the analyst returns the sample to the storage area.
- 9.12 **Sample Disposal.** Accutest retains all samples and sample extracts under proper storage for a maximum of 30 days following receipt. Longer storage periods are accommodated on a client specific basis if required. Samples may also be returned to the client for disposal.

Accutest disposes of all laboratory wastes following the requirements of the Resource Conservation and Recovery Act (RCRA). The Company has obtained and maintains an EPA waste generator identification number, CAL00218798

Sample management generates a sample disposal dump sheet from the LIMS tracking system as needed, which lists all samples whose holding period has expired.

Samples classified as PCB hazardous wastes are labeled and packaged according to the requirements in 40 CFR 761.

Laboratory wastes are collected by waste stream in designated areas throughout the laboratory. Waste streams are consolidated weekly and transferred to stream specific drums for disposal through a permitted waste management contractor.

All solvent extracts and digestates are collected for disposal following the thirty-day holding period and drummed according to their specific waste stream category. Chlorinated solvent extracts are drummed as chlorinated wastes (i.e., Methylene Chloride). Non-chlorinated solvent extracts are drummed as non-chlorinated wastes (i.e., acetone, hexane, methanol, and mixed solvents). Digestates are collected for disposal following the thirty-day holding period and drummed as corrosive liquid containing metals.

10.0 LABORATORY INSTRUMENTATION AND MEASUREMENT STANDARDS

Requirement: The laboratory has established procedures, which assure that instrumentation is performing to a pre-determined operational standard prior to the analysis of any samples. In general, these procedures follow the regulatory agency requirements established in promulgated methodology. The instrumentation selected to perform specified analysis are capable of providing the method specified uncertainty of measurement needed. These procedures are documented and incorporated into the standard operating procedures for the method being executed.

10.1 **Mass Tuning – Mass Spectrometers** The mass spectrometer tune and sensitivity is monitored to assure that the instrument is assigning masses and mass abundances correctly and that the instrument has sufficient sensitivity to detect compounds at low concentrations. This is accomplished by analyzing a specific mass tuning compound at a fixed concentration. If the sensitivity is insufficient to detect the tuning compound, corrective action must be performed prior to the analysis of standards or samples. If the mass assignments or mass abundances do not meet criteria, corrective action must be performed prior to the analysis of standards or samples.

10.2 **Wavelength Verification – Spectrophotometers** Spectrophotometer detectors are checked on a regular schedule to verify proper response to the wavelength of light needed for the test in use. If the detector response does not meet specifications, corrective action (detector adjustment or replacement) is performed prior to the analysis of standards or samples.

10.3 **Inter-element Interference Checks (Metals)** Inductively Coupled Plasma Emission Spectrophotometers (ICP) are subject to a variety of spectral interferences, which can be minimized or eliminated by applying interfering element correction factors and background correction points. Interfering element correction factors are checked on a specified frequency through the analysis of check samples containing high levels of interfering elements. Analysis of single element interferant solutions is also conducted at a specified frequency.

If the check indicates that the method criteria have not been achieved for any element in the check standard, the analysis is halted and data from the affected samples are not reported. Sample analysis is resumed after corrective action has been performed and the correction factors have been re-calculated.

New interfering element correction factors are calculated and applied whenever the checks indicate that the correction factors are no longer meeting criteria. At a minimum, correction factors are replaced once a year.

- 10.4 Calibration and Calibration Verification.** Many tests require calibration using a series of reference standards to establish the concentration range for performing quantitative analysis. Instrument calibration is performed using standards that are traceable to national standards. Method specific procedures for calibration are followed prior to any sample analysis.

Calibration is performed using a linear regression calculation, calibration factors calculated from the curve, or other curve fitting equations. The calibration must meet method specific criteria for linearity or precision. If the criteria are not achieved, corrective action (re-calibration or instrument maintenance) is performed. The instrument must be successfully calibrated before analysis of samples can be conducted.

Initial calibration for metals analysis performed using inductively coupled plasma (ICP) employs the use of a single standard and a calibration blank to establish linearity. The calibration blank contains all reagents that are placed into the calibration standard with the exception of the target elements. Valid calibration blanks must not contain any target elements.

Initial calibrations must be verified using a single concentration calibration standard from a second source. Second source standard is defined as a standard from a second or separate vendor. If no other vendor is available; then a second and separately prepared lot number from the primary vendor will suffice. The continuing validity of existing calibrations must be regularly verified using a single calibration standard. The response to the standard must meet pre-established criteria that indicate the initial calibration curve remains valid. If the criteria are not achieved corrective action (re-calibration) is performed before any additional samples may be analyzed.

Calibration verification is also performed whenever it appears that the analytical system is out of calibration or no longer meets the calibration requirements. It is also performed when the time period between calibration verifications has expired.

- 10.5 Linear Range Verification and Calibration (ICP).** Linear range verification is performed for all ICP instrumentation. The regulatory program or analytical method specifies the verification frequency. A series of calibration standards are analyzed over a broad concentration range. The data from these analyses are used to determine the valid analytical range for the instrument. ICP instrument calibration is routinely performed using a single standard at a concentration within the linear range and a blank.

Some methods or analytical programs require a low concentration calibration check to verify that instrument sensitivity is sufficient to detect target elements at the reporting limit. The analytical method or regulatory program defines the criteria used to evaluate the low concentration calibration check. If the low calibration check fails criteria, corrective action is performed and verified through reanalysis of the low concentration calibration check before continuing with the field sample analysis. .

- 10.6 **Retention Time Development and Verification (GC).** Chromatographic retention time windows are developed for all analysis performed using gas chromatographs with conventional detectors. An initial experimental study is performed, which establishes the width of the retention window for each compound. The retention time width of the window defines the time ranges for elution of specified target analytes on the primary and confirmation columns. Retention time windows are established upon initial calibration, applying the retention time range from the initial study to each target compound. Retention times are regularly confirmed through the analysis of an authentic standard during calibration verification. If the target analytes do not elute within the defined range during calibration verification, the instrument must be recalibrated and new windows defined. New studies are performed when major changes, such as column replacement are made to the chromatographic system.
- 10.7 **Equipment List.** See Appendix IV for a listing of all equipment used for measurement and/or calibration in laboratory processes.

11.0 INSTRUMENT MAINTENANCE

Requirement. Documented procedures have been established for conducting equipment maintenance. The procedure includes maintenance schedules if required or documentation of daily maintenance activities. All instrument maintenance activities are documented in instrument specific logbooks.

- 11.1 **Routine, Daily Maintenance.** Routine, daily maintenance is required on an instrument specific basis and is performed each time the instrument is used. Daily maintenance includes activities to insure a continuation of good analytical performance. This may include performance checks that indicate if non-routine maintenance is needed. If performance checks indicate the need for higher level maintenance, the equipment is taken out of service until maintenance is performed. Analysis cannot be continued until all performance checks meet established criteria and a return to operational control has been demonstrated and documented. The individual assigned to the instrument is responsible for daily maintenance.
- 11.2 **Non-routine Maintenance.** Non-routine maintenance is initiated for catastrophic occurrences such as instrument failure. The need for non-routine maintenance is indicated by failures in general operating systems that result in an inability to conduct required performance checks or calibration. Equipment in this category is taken out of service, tagged accordingly and repaired before attempting further analysis. Before initiating repairs, all safety procedures for safe handling of equipment during maintenance, such as lock-out/tag-out are followed. Analysis is not resumed until the instrument meets all operational performance checks criteria, is capable of being calibrated and a return to operational control has been demonstrated and documented. Section supervisors are responsible for identifying non-routine maintenance episodes and initiating repair activities to bring the equipment on-line. This may include initiating telephone calls to maintenance contractors if necessary. They are responsible for documenting all details related to the occurrence and repair.
- 11.3 **Scheduled Maintenance.** Modern laboratory instrumentation rarely requires regular preventative maintenance. If required, the equipment is placed on a schedule, which dictates when maintenance is needed. Examples include annual balance calibration by an independent provider or ICP preventative maintenance performed by the instrument manufacturer. Section supervisors are responsible for initiating scheduled maintenance on equipment in this category. Scheduled maintenance is documented using routine documentation practices.
- 11.4 **Maintenance Documentation.** Non-routine maintenance activities are documented in logbooks assigned to instruments and equipment used for analytical measurements. The logbooks contain preprinted forms, which specify the required maintenance activities. The analyst or supervisor performing or initiating the maintenance activity is required to check the activity upon its completion and initial the form. This includes documenting that the instrument has been returned to operational control following the completion of the activity.

12.0 QUALITY CONTROL PARAMETERS, PROCEDURES, AND CORRECTIVE ACTION

Requirement: All procedures used for test methods incorporate quality control parameters to monitor elements that are critical to method performance. Each quality parameter includes acceptance criteria that have been established by regulatory agencies for the methods in use. Criteria may also be established through the accumulation and statistical evaluation of internal performance data. Data obtained for these parameters during routine analysis must be evaluated by the analyst, and compared to the method criteria in use. If the criteria are not achieved, the procedures must specify corrective action and conformation of control before proceeding with sample analysis. QC parameters, procedures, and corrective action must be documented within the standard operating procedures for each method. In the absence of client specific objectives the laboratory must define qualitative objectives for completeness and representativeness of data.

- 12.1 **Procedure.** Bench analysts are responsible for methodological quality control and sample specific quality control. Each method specifies the control parameters to be employed for the method in use and the specific procedures for incorporating them into the analysis. These control parameters are analyzed and evaluated with every designated sample group (batch).

The data from each parameter provides the analyst with critical decision making information on method performance. The information is used to determine if corrective action is needed to bring the method or the analysis of a specific sample into compliance. These evaluations are conducted throughout the course of the analysis. Each control parameter is indicative of a critical control feature. Failure of a methodological control parameter is indicative of either instrument or batch failure. Failure of a sample control parameter is indicative of control difficulties with a specific sample or samples.

Sample Batch. All samples analyzed in the laboratory are assigned to a designated sample batch, which contains all required quality control samples and a defined maximum number of field samples that are prepared and/or analyzed over a defined time period. The maximum number of field samples in the batch is 20. ANC has incorporated the NELAP batching policy as the sample-batching standard. This policy incorporates the requirement for blanks and spiked blanks as a time based function as defined by NELAP. Accordingly, the specified time period for a sample batch is 24 hours. Matrix spike/matrix spike duplicate, matrix spikes and duplicates are defined as sample frequency based functions and may be applied to several batches until the frequency requirement has been reached. A matrix spike/matrix spike duplicate, matrix spikes and/or duplicate is required every 20 samples.

Client criteria that defines a batch as a time based function which includes a matrix spike/matrix spike duplicates as a contractual specification will be honored. The typical batch contains a blank and a laboratory control sample (LCS or spiked blank). Batch documentation includes lot specifications for all reagents and standards used during preparation of the batch.

- 12.2 **Methodological Control Parameters and Corrective Action.** Prior to the analysis of field samples the analyst must determine that the method is functioning properly. Specific control parameters indicate whether critical processes meet specified requirements before continuing with the analysis. Method specific control parameters must meet criteria before sample analysis can be conducted. Each of these parameters is related to processes that are under the control of the laboratory and can be adjusted if out of control.

Method Blank. A method blank is analyzed during the analysis of any field sample. The method blank is defined as a sample. It contains the same standards (internal standards, surrogates, matrix modifiers, etc.) and reagents that are added to the field sample during analysis, with the exception of the sample itself. If the method blank contains target analyte(s) at concentrations that exceed reporting limit concentrations and is greater than 1/10 of the amount measured in any sample, the source of contamination is investigated and eliminated before proceeding with sample analysis. Systematic contamination is documented for corrective action and resolved following the established corrective action procedures.

Blank Spikes (LCS). A laboratory control sample (spiked blank or commercially prepared performance evaluation sample) is analyzed along with field samples to demonstrate that method accuracy is within acceptable limits. These spike solutions may be from different sources than the sources of the solutions used for method calibration depending upon the method requirements. The performance limits are derived from published method specifications or from statistical data generated from the analysis of laboratory method performance samples. Spiked blanks are blank matrices (reagent water or clean sand) spiked with target parameters and analyzed using the same methods used for samples. Accuracy data is compared to laboratory derived limits to determine if the method is in control. Laboratory control samples (LCS) are commercially prepared spiked samples in an inert matrix. Performance criteria for recovery of spiked analytes are pre-established by the commercial entity preparing the sample. The sample is analyzed in the laboratory as an external reference.

Accuracy data is compared to the applicable performance limits. If the spike accuracy exceeds the performance limits, corrective action, as specified in the SOP for the method is performed and verified before continuing with a field sample analysis.

Blanks and spikes are routinely evaluated before samples are analyzed. However, in situations where sample analysis is performed using an autosampler, they may be evaluated after sample analysis has occurred. If the blanks and spikes do not meet criteria, sample analysis is repeated.

Proficiency Testing. Proficiency test samples (PTs) are single or double blind spikes, introduced to the laboratory to assess method performance. PTs may be introduced as double blinds submitted by commercial clients, single or double blinds from regulatory agencies, or internal blinds submitted by the QA group.

A minimum of two single blind studies must be performed each year for every parameter in aqueous and solid matrices for each field of testing for which the laboratory maintains

accreditation. Proficiency samples must be purchased as blinds from an A2LA accredited vendor. Data from these studies are provided to the laboratory by the vendor and reported to accrediting agencies. If unsatisfactory performance is noted, corrective action is performed to identify and eliminate any sources of error. A new single blind must be analyzed if required to demonstrate continuing proficiency.

PT samples performed for accrediting agencies or clients, which do not meet performance specifications, require a written summary that documents the corrective action investigation, findings, and corrective action implementation.

Single or double blind proficiency test samples may be employed for self-evaluation purposes. Data from these analyses are compared to established performance limits. If the data does not meet performance specifications, the system is evaluated for sources of acute or systematic error. If required, corrective action is performed and verified before initiating or continuing sample analysis.

Trend Analysis for Control Parameters. The quality assurance staff is responsible for continuous analytical improvement through quality control data trend analysis. Accuracy data for spiked parameters in the spiked blank are statistically evaluated daily for trends indicative of systematic problems. Data from LCS parameters and surrogates are pooled on a method, matrix, and instrument basis. This data is evaluated by comparison to existing control and warning limits. Trend analysis is performed automatically as follows:

- Any point outside the control limit
- Any three consecutive points between the warning and control limits
- Any eight consecutive points on the same side of the mean.
- Any six consecutive points increasing or decreasing

12.3 Sample Control Parameters and Corrective Action The analysis of samples can be initiated following a successful demonstration that the method is operating within established controls. Additional controls are incorporated into the analysis of each sample to determine if the method is functioning within established specifications for each individual sample. Sample QC data is evaluated and compared to established performance criteria. If the criteria are not achieved the method or the SOP specifies the corrective action required to continue sample analysis. In many cases, failure to meet QC criteria is a function of sample matrix and cannot be remedied. Each parameter is designed to provide quality feedback on a defined aspect of the sampling and analysis episode.

Duplicates. Duplicate sample analysis is used to measure analytical precision. This can also be equated to the samples homogeneity. Precision criteria are method dependent. If precision criteria are not achieved, corrective action or additional action may be required. Recommended action must be completed before sample data can be reported.

Laboratory Spikes & Spiked Duplicates. Spikes and spiked duplicates are used to measure analytical precision and accuracy for the sample matrix selected. Precision and accuracy criteria are method dependent. If precision and accuracy criteria are not achieved, corrective action or additional action may be required. Recommended action must be completed before reporting sample data.

Serial Dilution (Metals). Serial dilutions of metals samples are analyzed to determine if analytical matrix effects may have impacted the reported data. If the value of the serially diluted samples does not agree with the undiluted value within a method-specified range, the sample matrix may be causing interferences, which may lead to either a high or low bias. If the serial dilution criterion is not achieved, it must be flagged to indicate possible bias from matrix effects.

Post Digestion Spikes. Digested samples are spiked and analyzed to determine if matrix interferences are biasing the results when the pre-digestion spike (matrix spike) recovery falls outside the control limits. It may also be used to determine potential interferences per client's specification. The sample is spiked at the concentration specified in the method SOP. No action is necessary if the post digestion spike is outside of the method criteria, unless a preparation problem is suspected with the spike, in which case the post digestion spike should be re-prepared and reanalyzed.

Surrogate Spikes (Organics). Surrogate spikes are organic compounds that are similar in behavior to the target analytes but unlikely to be found in nature. They are added to all quality control and field samples to measure method performance for each individual sample. Surrogate accuracy limits are derived from published method specifications or from the statistical evaluation of laboratory generated surrogate accuracy data. Accuracy data is compared to the applicable performance limits. If the surrogate accuracy exceeds performance limits, corrective action, as specified in the method or SOP is performed before sample data can be reported.

Internal Standards (Organic Methods). Internal standards are retention time and instrument response markers added to every sample to be used as references for quantitation. Their response is compared to reference standards and used to evaluate instrument sensitivity on a sample specific basis. Internal standard retention time is also compared to reference standards to assure that target analytes are capable of being located by their individual relative retention time.

If internal standard response criteria are not achieved, corrective action or additional action may be required. The recommended action must be completed before sample data can be reported.

If the internal standard retention time criteria are not achieved corrective action or additional action may be required. This may include re-calibration and re-analysis. Additional action must be completed before sample data is reported.

- 12.4 **Laboratory Derived Quality Control Criteria.** Control criteria for in-house methods and client specific modifications that exceed the scope of published methodology are defined and documented prior to the use of the method. The Quality Assurance Director is responsible for identifying additional control criteria needs. Control parameters and criteria, based on best technical judgment are established using input provided by the operations staff. These control parameters and criteria are documented and incorporated into the method.

The laboratory-derived criteria are evaluated for technical soundness on spiked samples prior to the use of the method on field samples. The technical evaluation is documented and archived by the Quality Assurance Staff.

When sufficient data from the laboratory developed control parameter is accumulated, the data is statistically processed and the experimentally derived control limits are incorporated into the method.

- 12.5 **Bench Review & Corrective Action.** The bench chemists are responsible for all QC parameters. Before proceeding with sample analysis, they are required to successfully meet all instrumental QC criteria. They have the authority to perform any necessary corrective action before proceeding with sample analysis. Their authority includes the responsibility for assuring that departures from documented policies and procedures do not occur.

The bench chemists are also responsible for all sample QC parameters. If the sample QC criteria are not achieved, they are authorized and required to perform the method specified corrective action before reporting sample data.

- 12.6 **Data Qualifiers.** An alpha character coding system is employed for defining use limitations for reported data. These limitations are applied to analytical data by the analyst to clarify the usefulness of the reported data for data user. Common data qualifiers and their definitions are as follows:

Organics.

- J: Indicates an estimated value. Applied to calculated concentrations for tentatively identified compounds and qualitatively identified compounds whose concentration is below the reporting limit, but above the MDL.
- N: Indicates qualitative evidence of a tentatively identified compound whose identification is based on a mass spectral library search and is applied to all TIC results.
- C: Applied to pesticide data that has been qualitatively confirmed by GC/MS.
- B: Used for analytes detected in the sample and its associated method blank.
- E: Applied to compounds whose concentration exceeds the upper limit of the calibration range.

Metals and Inorganics.

- J: Applied if the reported concentration value was less than the reporting limit but greater than the MDL.
- E: Estimated concentration caused by the presence of interferences, normally applied when the serial dilution is out.
- ***: Spike sample recovery not within control limits, or Duplicate or matrix spike duplicate analysis not within control limits.

12.7 QA Monitoring. The Reporting staff conducts a spot review of completed data packages prior to client release for specified projects. This review includes an examination of QC data for compliance. If non-conformances are detected, the reporting staff places an immediate stop on the release of the data and initiates corrective action to rectify the situation. The data package is released when the package becomes compliant with all quality requirements. If compliance is not possible, the data is qualified and an appropriate case narrative is generated for inclusion in the data package.

If the review reveals trends indicative of systematic problems, QA initiates an investigation to determine the cause. If process defects are detected, a corrective action is implemented and monitored for effectiveness.

Performance Limits. The Quality Assurance Director is responsible for compilation and maintenance of all precision and accuracy data used for performance limits. Quality control data for all test methods are accumulated and stored in the laboratory information management system (LIMS). Parameter specific QC data is extracted annually and statically processed to develop laboratory specific warning limits and control limits. The new limits are reviewed and approved by the supervisory staff prior to their use for data assessment. The new limits are used to evaluate QC data for compliance with method requirements for a period of one year. Laboratory generated limits appear on all data reports.

12.8 Data Package Review. Accutest employs multiple levels of data review to assure that reported data has satisfied all quality control criteria and that client specifications and requirements have been met. Each production department has developed specific data review procedures, which must be completed before data is released to the client.

Analytical Review. The analyst conducts the primary review of all data. This review begins with a check of all instrument and method quality control and progresses through sample quality control, concluding with a check to assure that the client's requirements have been executed. Analyst checks focus on a review of qualitative determinations and checks of precision and accuracy data to verify that existing laboratory criteria have been achieved. Checks at this level may include comparisons with project specific criteria if applicable. The analyst has the authority and responsibility to perform corrective action for any out-of-control parameter or nonconformance at this stage of review.

Analysts who have met the qualification criteria for the method in use perform secondary, peer level data reviews. Analyst qualification requirements include a valid demonstration of

capability and demonstrated understanding of the method SOP. Section supervisors may perform secondary review in-lieu of a peer review. Supervisors review 100% of the data produced by their department. It includes a check of all manual calculations; an accuracy check of manually transcribed data from bench sheets to the LIMS, a check of calibration and continuing calibration, all QC criteria and a comparison of the data package to client specified requirements. Also included are checks to assure the appropriate methodology was applied and that all anomalous information was properly flagged for communication in the case narrative. Supervisors have the authority to reject data and initiate re-analysis, corrective action, or reprocessing.

All laboratory data requiring manual entry into LIMS system is double-checked by the analysts performing initial data entry and the section supervisor. Verification of supervisory review is indicated on the raw data summary by the supervisor's initials and date.

Hard copies of manually integrated chromatographic peaks are printed that clearly depict the manually drawn baseline. The hard copy is reviewed and approved by the section supervisor (initialed and dated) and included in the data package of all full tier reports or the archived batch records of commercial report packages.

A manager or supervisor only has permission to edit electronic data that has been committed to the LIMS. These edits may be required if needs for corrections are indicated during the final review. A GALP audit record for all electronic changes in the LIMS is automatically appended to the record.

The group manager performs a tertiary review on a spot check basis. This review includes an evaluation of QC data against acceptance criteria and a check of the data package contents to assure that all analytical requirements and specifications were executed.

Report Generation Review. The report generation group reviews all data and supporting information delivered by the laboratory for completeness and compliance with client specifications. Missing deliverables are identified and obtained from the laboratory. The group also reviews the completed package to verify that the delivered product complies with all client specifications. Non-analytical defects are corrected before the package is sent to the client.

Project Management/Quality Control Review. Spot-check data package reviews are performed by the project management staff. Project management reviews focus on project specifications. If the project manager identifies defects in the product prior to release, he initiates immediate corrective action to rectify the situation.

Data Reporting. Analytical data is released to clients following a secondary review by the group supervisor. Hard copy support data is compiled by the report generation group and assembled into the final report. The report is sent to the client following reviews by the report generation staff.

All data reports include specified information, which is required to identify the report and its contents. This information includes a title, name and address of the laboratory, a unique report number, total number of pages, clients name and address, analytical method identification, arriving sample condition, sample and analysis dates, test results with units of measurement, authorized signature of data release, and NELAC requirements certification.

- 12.9 **Electronic Data Reduction.** Raw data from sample analysis is entered into the laboratory information management system (LIMS) using automated processes or manual entry. Final data processing is performed by the LIMS using procedures developed by the Company.

All LIMS programs are tested and validated prior to use to assure that they consistently produce correct results. The Information Technology Staff performs software validation testing. The testing procedures are documented in an SOP. Software programs are not approved for use until they have demonstrated that they are capable of performing the required calculations.

- 12.10 **Representativeness.** Data representativeness is based on the premise that qualitative and quantitative information developed for field samples is characteristic of the sample that was collected by the client and analyzed in the laboratory. The laboratory objective for representativeness defines data as representative if the criteria for all quality parameters associated with the analysis of the sample are achieved.

- 12.11 **Comparability.** Analytical data is defined as comparable when data from a sample set analyzed by the laboratory is representatively equivalent to other sample sets analyzed separately regardless of the analytical logistics. The laboratory will achieve 100% comparability for all sample data which meets the criteria for the quality parameters associated with its analysis using the method requested by the client.

13.0 CORRECTIVE ACTION SYSTEM (Incident Reporting)

Requirement. The laboratory employs a procedure for correcting defective processes, safety issues, efficiency issues, systematic errors, and quality defects enabling the staff to systematically improve product quality. The system includes procedures for communicating items requiring corrective action to responsible individuals, corrective action tracking procedures, corrective action documentation, monitoring of effectiveness, and reports to management. The system is fully documented in a standard operating procedure. Individual corrective actions and responses are documented in a dedicated database.

- 13.1 **Procedure.** Corrective action is the step that follows the identification of a process defect. The type of defect determines the level of documentation, communication, and training necessary to prevent re-occurrence of the defect or non-conformance. The formal system is maintained by the quality assurance department. Operations management is responsible for working within the system to resolve identified deficiencies.

Routine Corrective Action. Routine corrective action is defined as the procedures used to return out of control analytical systems back to control. This level of corrective action applies to all analytical quality control parameters or analytical system specifications.

Bench analysts have full responsibility and authority for performing routine corrective action. The resolution of defects at this level does not require a procedural change or staff re-training. The analyst is free to continue work once corrective action is complete and the analytical system has been returned to control. Documentation of routine corrective actions is limited to logbook comments for the analysis being performed.

Non-routine corrective action. Non-routine corrective action applies to situations where the bench analysts failed to perform routine corrective action before continuing analysis. Supervisors and Department Managers perform corrective action in these situations. Documentation of all non-routine corrective actions is performed using the corrective action system.

Turnbacks. Turnbacks are defined as anything that stops your work, but does not affect quality. These efficiency issues are tracked in the incident reporting system, but do not need immediate corrective action. Repeat turnbacks may result in their status changing to a corrective action worthy of Supervisor/Manager attention and a process change. Process change requires development, documentation, planning, implementation and training.

- 13.2 **Documentation & Communication.** Routine corrective actions are documented as part of the analytical record. Notations are made in the comments section of the analytical chronicle or data sheet detailing the nonconformance and corrective action. Continuation of the analysis indicates that return to control was successful.

Corrective actions for process changes are documented, tracked and monitored for effectiveness. Supervisors or senior staff members may initiate corrective actions by generating an Incident Reporting System.

The Incident Reporting System is an Adobe application. All Employees' have a short-cut on their computer desk-top to the Incident Reporting Form. The initiator fills out the form and the form is sent to the Lab Director via E-mail. The IR Form is reviewed and it is determined if the incident is a turnback, or a Non-Routine Corrective Action. Turnbacks are summarized for the monthly management meeting. Non-Routine Corrective Actions are sequentially numbered, placed in the NCAR_In_Process directory by years and forwarded by Email to the responsible parties and the QA Officer.

The responsible party identifies the root cause of the defect, initiates the immediate fix and develops and implements the procedural change. Existing documentation such as SOPs are edited to reflect the change. The affected staff is informed of the procedural change through a formal training session. The training is documented and copies are placed into individual training files. The corrective action form is completed by the responsible party and returned to the QAO. All relevant information for the NCCAR is stored in the directory NCAR_Completed.

Monitoring. The QA Staff monitors the implemented corrective action until it is evident that the action has been effective and the defect has been eliminated. The corrective actions are updated by QA to reflect closure of the corrective action. The QA staff assigns a number to the corrective action tracking. Additional monitoring of the corrective action may be conducted during routine laboratory audits.

If QA determines that the corrective action response has not effectively remedied the deficiency, the process continues with a re-initiation of the corrective action. Corrective action continues until the defect is eliminated. If another procedural change is required, it is treated as a new corrective action, which is documented and monitored using established procedures.

Client Notification. Defective processes, systematic errors, and quality defects, detected during routine audits may have negative impacts on data quality. In some cases, data that has been released to clients may be affected. If defective data has been released for use, ANC will notify the affected clients of the defect and provide specific details regarding the magnitude of the impact to their data.

14.0 PROCEDURES FOR EXECUTING CLIENT SPECIFICATIONS

Requirement. Systems have been established for evaluating and processing client specifications for routine and non-routine analytical services. The systems enable the client services staff to identify, evaluate, and document the requested specifications to determine if adequate resources are available to perform the analysis. The system includes procedures for communicating the specifications to the laboratory staff for execution and procedures for verifying the specifications have been executed.

- 14.1 **Client Specific Requirements.** The project manager is the primary contact for clients requesting laboratory services. Client specifications are communicated using several mechanisms. The primary sources of information are the client's quality assurance project plan (QAPP) and the analytical services contract both of which detail the analytical, quality control and data reporting specifications for the project. In the absence of a QAPP, projects specifications can also be communicated using contracts, letters of authorization, or letters of agreement, which may be limited to a brief discussion of the analytical requirements and the terms and conditions for the work. These documents may also include pricing information, liabilities and scope of work, in addition to the analytical requirements. QAPPs include detailed analytical requirements and data quality objectives, which supersede those found in the referenced methods. This information is essential to successful project completion.

The client services staff provides additional assistance to clients who are unsure of the specifications they need to execute the sampling and analysis requirements of their project. They provide additional support to clients who require assistance in results interpretation as needed, provided they possess the expertise required to render an opinion.

The project manager is responsible for obtaining project documents, which specify the analytical requirements. Following project management review, copies are distributed to the QA Director and the appropriate departmental managers for review and comment. The original QAPP is filed in a secure location.

- 14.2 **Requirements for Non-Standard Analytical Specifications.** Client requirements that specify departures from documented policies, procedures, or standard specifications must be submitted to Accutest in writing. These requirements are reviewed and approved by the technical staff before the project is accepted. Once accepted, the non-standard requirements become analytical specifications, which follow the routine procedure for communicating client specifications. Specific Client Specification are filed in the Project Management Directory. Departures from documented policies, procedures, or standard specifications that do not follow this procedure are not permitted.

- 14.3 **Evaluation of Resources.** A resource evaluation is completed prior to accepting projects submitted by clients. The evaluation is initiated by the client services staff who prepares a brief synopsis that includes the logistical requirements of the project. Logistical specifications for new projects are summarized in writing for evaluation by the affected departments. The specifications are evaluated by the department manager from a scheduling and hardware resources perspective. The project is not accepted unless the department managers have the necessary resources to execute the project according to client specifications.
- 14.4 **Documentation.** New projects are initiated using a project set up form, which is completed prior to the start of the project. This form details all of the information needed to correctly enter the specifications for each client sample into the laboratory information management system (LIMS). The form includes data reporting requirements, billing information; data turnaround times, QA level, state of origin, and comments for detailing project specific requirements. The project manager is responsible for obtaining this information from the client and completing the form prior to sample arrival and login. For less complicated requirements or unscheduled work; the chain of custody will contain all the necessary information.

Sample receipt triggers project creation and the login process. The information on the set-up form is entered into the LIMS immediately prior to logging in the first sample. The set up form may be accompanied by a quotation, which details the analytical product codes and sample matrices. These details are also entered into the LIMS during login.

Special information is distributed to the laboratory supervisors and login department in electronic or hardcopy format upon project setup. All, project specific information is retained on the file server.

Job Level Documentation:

\\ancdoc\Documents\Disk1\coc

Account and Project Level Documentation

\\Accunca.accutest.com\depts\project_mgt

All client communications; regarding a job, project or account is documented either via email and notes stored as .pdfs in the directories above.

Department managers prepare summary sheets that detail client specific analytical requirements for each test. Bench analysts use these sheets to obtain information regarding client specific analytical requirements before analyzing samples. A program code is established for each client that links the client specifications to a client project. This code is attached to a project by the project manager at login and listed on the work list for each work group conducting analysis for clients with standing requirements.

- 14.5 **Communication.** A pre-project meeting is held between client services and the operations managers to discuss the specifications described in the QAPP, contract and/or related

documents. Project logistics are discussed and finalized and procedures are developed to assure proper execution of the client's analytical specifications and requirements. Questions, raised in the review meeting, are discussed with the client for resolution. Exceptions to any requirements, if accepted by the client, are documented and incorporated into the QAPP or project documentation records.

Non-standard specifications for individual clients are documented in the LIMS at the client account level or program level. Simple specifications are documented as comments for each project. Once entered into the LIMS, these specifications become memorialized for all projects related to the client account. Complex specifications are assigned program codes that link the specification to detailed analytical specifications.

Specifications that are not entered into the LIMS are prohibited unless documented in an interdepartmental memo, which clearly identifies the project, client and effective duration of the specification.

- 14.6 **Operational Execution.** A work schedule (WIP) is prepared for each analytical department on a daily basis. Analytical specifications or program codes from recently arrived samples have now been entered into the LIMS database. The database is sorted by analytical due date and holding time, into product specific groups. Samples are scheduled for analysis by due date and holding time. The completed schedule, which is now defined as a work list, is printed. The list contains the client requested product codes, program codes and specifications required for the selected sample(s). Special requirements are communicated to the analyst using the comments section or relayed through verbal instructions provided by the supervisor. The bench analyst assumes full responsibility for performing the analysis according to the specifications printed on the work sheet.
- 14.7 **Verification.** Prior to the release of data to the client, laboratory section managers and the report generation staff review the report and compare the completed product to the client specifications documentation to assure that all requirements have been met. Project managers perform a spot check of projects with unique requirements to assure that the work was executed according to specifications.

15. CLIENT COMPLAINT RESOLUTION PROCEDURE

Requirement. The laboratory uses the Incident Reporting System to investigate and manage client complaints. The system includes procedures for documenting the complaint and communicating it to the appropriate department for resolution. The system also includes a quality assurance evaluation to determine if the complaint is related to systematic defects requiring corrective action and process changes.

- 15.1 **Procedure.** Client complaints are entered into the Incident Report System by any member of the staff. The report is sent directly to the Laboratory Director and the QA Officer. Reports are assigned to the responsible departments for resolution. The resolution is reviewed by quality assurance (QA) and the originator, then communicated to the client. QA reviews the complaint and resolution to determine if systematic defects exist. If no systemic defects are present and the proposed resolution is sufficient, QA will close the complaint/inquiry with a No Further Action necessary. If systematic defects are present, QA initiates an additional incident report for the responsible party who develops and implements a response that eliminates the defect.
- 15.2 **Documentation.** A record of conversation is maintained within the PDF Incident Report. The message is distributed to the QA staff and the party bearing responsibility for resolution by E-Mail. The complaint resolution is documented on the message by the responsible party and returned to the originator. A copy is sent to QA for review.
- 15.3 **Corrective Action.** Responses to data queries are required from the responsible party. At a minimum, the response addresses the query and provides an explanation to the complaint. Formal corrective action may focus on the single issue expressed in the complaint. Corrective action may include reprocessing of data, editing of the initial report, and re-issue to the client. If the QA review indicates a systematic error, process modification is required. The defective process at the root of the complaint is changed. SOPs are either created or modified to reflect the change. The party responsible for the process implements process changes.
- 15.4 **QA Monitoring.** Process changes, implemented to resolve systematic defects, are monitored for effectiveness by QA. If monitoring indicates that the process change has not resolved the defect, QA works with the department management to develop and implement an effective process. If monitoring indicates that the defect has been resolved, monitoring is slowly discontinued and the corrective action is closed. Continued monitoring is incorporated as an element of the annual system audit.

16.0 CONTROL OF NONCONFORMING PRODUCT

Requirement: Policies and procedures have been developed and implemented that describe the procedures employed by the laboratory when any aspect of sample analysis or data reporting do not conform to established procedures or client specifications. These procedures include steps to ensure that process defects are corrected and affected work is evaluated to assess its impact to the client. Non-conforming issues are also handled in the Incident Reporting system.

- 16.1 **Procedure:** Nonconforming product is identified through routine internal review and audit practices or through client inquiry. The individuals who identify the nonconformance or receiving a nonconformance inquiry immediately initiate an Incident Report informing the Laboratory Director and the Quality Assurance Director. The Laboratory Director initiates an evaluation of the nonconformance through the Quality Assurance Department and takes full responsibility for managing the process and identifying the course of action to take, initiating corrective action and mitigating the impact of the nonconformance to the client.

- 16.2 **Corrective Action:** The outcome of the evaluation dictates the course of action. This includes client notification when the quality of data reported has been impacted and may also include corrective action if applicable. However, additional action may be required including cessation of analysis and withholding and or recalling data reports. If the evaluation indicates that nonconforming data may have been issued to clients, the client is immediately notified and data may be recalled. If work has been stopped because of a nonconformance, the Laboratory Director is the only individual authorized to direct a resumption of analysis.

Nonconformance caused by systematic process defects require retraining of the personnel involved as an element of the corrective action solution.

- 16.3 **Data Inquiry Program:** A program contained within the Corrective action program used to capture, address, and respond to client challenges to our sample results or data package components. A Complaint or Data Inquiry is begun by using the Initiate icon. This leads to an interactive template which allows the user to enter an Accutest Job number if it is a data inquiry. If it is a complaint unrelated to an Accutest job, the program will open directly to the template requiring the user to populate any number of the program fields. If using the Data Inquiry entry point along with an Accutest job number, the program will automatically populate some of the fields by extracting the information from LIMS.

17.0 CONFIDENTIALITY PROTECTION PROCEDURES

Requirements: Policies and procedures have been developed to protect client data from release to unauthorized parties or accidental release of database information through accidental electronic transmission or illegal intrusion. These policies have been communicated to clients and staff. Electronic systems are regularly evaluated for effectiveness.
(SOP PM004) -Confidentiality Protection Procedures

- 17.1 **Client Anonymity.** Information related to the Company's clients is granted to employees on a "need to know" basis. An individual's position within the organization defines his "need to know". Individuals with "need to know" status are given password access to systems that contain client identity information and access to documents and document storage areas containing client reports and information. Access to client information by individuals outside of the Company is limited to the client and individuals authorized by the client.

Individuals outside of the Company may obtain client information through subpoena issued by a court of valid jurisdiction. Clients are informed when subpoenas are received ordering the release of their information.

Client information may be released directly to regulatory agencies without receiving client authorization under specified circumstances. These circumstances require that the regulatory agency have statutory authority under the regulations for laboratory certification and that ANC's operations fall under the purview of the regulation. In these situations, Accutest will inform the client of the regulatory agencies request for information pertaining to his data and proceed with the delivery of the information to the regulatory agency.

- 17.2 **Documents.** Access to client documents is restricted to employees in need to know positions. Copies of all client reports are stored in secure electronic archives with restricted access. Reports and report copies are distributed to individuals who have been authorized by the client to receive them. Data reports or data are not released to third parties without verbally expressed or written permission from the client.

17.3 **Electronic Data.**

Database Intrusion. Direct database entry is authorized for employees of Accutest only on a need to know basis. Entry to the database is restricted through a user specific multiple password entry system. Direct access to the database outside of the facility is possible through a dial-up connection. A unique password is required for access to the local area network. A second unique password is required to gain access to the database. The staff receives read or write level authorization on a hierarchical privilege basis.

- 17.4 **Information Requests.** Client specific data or information is not released to third parties without verbally expressed or written permission from the client. Written permission is required from third parties, who contact the Company directly for the release of information. Verbal requests will be honored only if they are received directly from the client. These requests must be documented in a record of communication maintained by the authorized recipient.
- 17.5 **Transfer of Records.** Archived data, which has previously been reported and transmitted to clients, is the exclusive property of Accutest Laboratories. In the event of a cessation of business activities due to business failure or sale, The Company's legal staff will be directed to arrange for the final disposition of archived data.

The final disposition of archived data will be accomplished using the approach detailed in the following sequence:

1. All data will be transferred to the new owners for the duration of the required archive period as a condition of sale.
2. If the new owners will not accept the data or the business has failed, letters will be sent to clients listed on the most recent active account roster offering them the option to obtain specific reports (identified by Accutest Job Number) at their own expense.
3. A letter will be sent to the NELAC accrediting authority with organizational jurisdiction over the company offering them the option to obtain all unclaimed reports at their own expense.
4. All remaining archived data will be recycled using the most expedient means possible.

18.0 QUALITY AUDITS AND SYSTEM REVIEWS

Requirement: The quality assurance group conducts regularly scheduled audits of the laboratory to assess compliance with quality system requirements, technical requirements of applied methodology, and adherence to documentation procedures. The information gathered during these audits is used to provide feedback to senior management and perform corrective action where needed for quality improvement purposes.

- 18.1 **Quality System Reviews.** Quality system reviews are performed annually by the Quality Assurance Director for the Company President. In this review, the laboratory is evaluated for compliance with the laboratory Quality Systems Manual (QSM) and the quality system standards of the National Environmental Laboratory Accreditation Conference. Findings, which indicate non-compliance or deviation from the QSM, are flagged for corrective action. Corrective actions require either a return to compliance or a plan change to reflect an improved quality process. The Quality Assurance Director is responsible for making and documenting changes to the QSM. These changes are reviewed by the Company President and The Laboratory Director prior to the approval of the revised system.
- 18.2 **Quality System Audits.** Quality system audits are conducted to evaluate the effectiveness and laboratory compliance with individual quality system elements. These audits are conducted on an established schedule. Audit findings are documented and communicated to the management staff and entered into the corrective action system for resolution. If necessary, retraining is conducted to assure complete understanding of the system requirements.
- 18.3 **Test Method Assessments.** Test Method Assessments are performed throughout the year following an established schedule. Selected analytical procedures are evaluated for compliance with standard operating procedures (SOPs) and method requirements. If non-conformances exist, the published method serves as the standard for compliance. SOPs are edited for compliance if the document does not reflect method requirements. Analysts are trained to the new requirements and the process is monitored by quality assurance. Analysts are retrained in method procedures if an evaluation of bench practices indicates non-compliance with SOP requirements.
- 18.4 **Documentation Audits.** Documentation audits are conducted annually. This audit includes a check of measurement processes that require manual documentation. It also includes checks of data archiving systems and a search to find and remove any inactive versions of SOPs that may still be present in the laboratory and being accessed by the analysts. Non-conformances are corrected on the spot. Procedural modifications are implemented if the evaluation indicates a systematic defect.
- 18.5 **Corrective Action Monitoring.** Defects or non-conformances that are identified during client or internal audits are documented in the corrective action systems and corrected through process modifications and/or retraining. Once a corrective action has been designed and

implemented, it is monitored for compliance on a regular basis by the QA staff. Spot corrections are performed if the staff is not following the new procedure. Monitoring of the corrective action continues until satisfactory implementation has been verified.

- 18.6 **Preventive Action.** Laboratory systems or processes, which may be faulty and pose the potential for nonconformances, errors, confusing reports or difficulties establishing traceability may be identified during internal audits. These items are highlighted for systematic change using the corrective action/incident reporting system and managed to .
- 18.7 **Client Notification.** Defective processes, systematic errors, and quality defects, detected during routine audits may have negative impacts on data quality. In some cases, data that has been released to clients may be affected. If defective data has been released for use, Accutest will immediately notify the affected clients of the defect and provide specific details regarding the magnitude of the impact to their data.
- 18.8 **Management Reports.** Formal reports of all audit and proficiency testing activity are prepared for the management staff and presented as they occur. Additional reports may be presented orally at regularly scheduled staff meetings

Management reports may also address the following topics:

- Status and results of internal and external audits,
- Status and results of internal and external proficiency testing,
- Identification of quality control problems in the laboratory,
- Discussion of corrective action/incident reporting program issues,
- Status of external certifications and approvals,
- Status of staff training and qualifications,
- Discussion of new quality system initiatives.
- Recommendations for further action on listed items are included in the report.

19.0 HEALTH AND SAFETY

Requirement. The company operates a formal health and safety program that complies with the requirements of the Occupational Health and Safety Administration. The program consists of key policies and practices that are essential to safe laboratory operation. All employees are required to receive training on the program elements. Job specific training is conducted to assure safe practices for specific tasks. All employees are required to participate in the program, receive initial and annual training, and comply with the program requirements. All plan and program requirements are detailed in the Health and Safety Program Manual.

- 19.1 **Policy.** Accutest Laboratories – Northern California will provide a safe and healthy working environment for its employees and clients while protecting the public and preserving the Company's assets and property. The company will comply with all applicable government regulations pertaining to safety and health in the laboratory and the workplace.

The objective of the Accutest-Northern California Health and Safety Program is to promote safe work practices that minimize the occurrence of injuries and illness to the staff through proper health and safety training, correct laboratory technique application and the use of engineering controls.

- 19.2 **Responsibilities.** The Health and Safety Program assists managers, supervisors and non-supervisory employees in control of hazards and risks to minimize the potential for employee and client injuries, damage to client's property and damage or destruction to ANC's facility.

The Health, Safety and Facilities Manager is responsible for implementing the Program's elements and updating its contents as necessary. He also conducts periodic audits to monitor compliance and assess the program's effectiveness. The Health, Safety and Facilities Manager is also responsible for creating and administering safety training for all new and existing employees.

The employee is responsible for following all safety rules established for their protection, the protection of others and the proper use of protective devices provided by the Company. The employee is also expected to comply with the requirements of the program at all times. Department Managers and Supervisors are responsible for ensuring the requirements of the Safety Program are practiced daily. The Company President retains the ultimate responsibility for the program design and implementation.

- 19.3 **Program Elements.** The Accutest Health and Safety Program consist of key program elements that compliment the company's health and safety objective. These elements form the essence of the health and safety policy and assure that the objectives of the program are achieved.

Safety Education and Training and Communication. Training is conducted to increase the staff's awareness of laboratory hazards and their knowledge of the safety practices and procedures required to protect them from those hazards. It is also used to communicate general safety procedures required for safe operation in a chemical laboratory.

Initial health and safety training for new employees is conducted during orientation. The training focuses on the Accutest Safety and Health Program and includes specific training for the hazards that may be associated with the employees duties. Training is also conducted for all program elements focusing on general, acceptable, laboratory safety procedures. Targeted training is conducted to address hazards or safety procedures that are specific to individual employee's work assignments. All training activities are documented and archived in individual training folders. A health and safety training inventory is maintained in the training database.

Safety Committee. The safety committee provides the employee with an opportunity to express their views and concerns on safety issues in a forum where those concerns will be addressed. This committee meets monthly to assure that the interests of the company and the well being of the employee are protected. They also serve as a catalyst for elevating the level of safety awareness among their peers.

Hazard Identification and Communication. The hazard communication program enables employees to readily identify laboratory hazards and the procedures to protect themselves from those hazards. This program complies with OSHA's Hazard Communication Standard, Title 29 Code of Federal Regulations 1910.1200 that requires the company to adopt and adhere to the following key elements:

- ◆ Material Safety Data Sheets (MSDS) must be available to any employee wishing to view them,
- ◆ The Company must maintain a Hazardous Chemicals Inventory (by location), which is updated on an annual basis,
- ◆ Containers are properly labeled,
- ◆ All employees must be provided with annual Hazard Communication and Right to Know training,

The hazard communication program also complies with the requirements of the New Jersey Worker and Community Right to Know Law, NJAC 8:95.

Identification of Workplace Hazards. The workplace hazard identification procedures have been designed to assure that hazards that have the potential to cause personnel injury or destruction of property are identified, managed and/or systematically eliminated from the operation. This system eliminates hazards, limits the potential for injury and increases the overall safety of the work environment.

Employee Exposure Assessment. Employee exposure assessment is performed to identify and evaluate potential exposure hazards associated with the employees work station. The exposure assessment data is used to determine if changes or modifications to the work station are needed to limit exposure to laboratory conditions that could negatively affect an employee's existing medical conditions.

Bloodborne Pathogens. Accutest has implemented the OSHA Bloodborne Pathogen Standard, 29CFR1910.1030 to reduce occupational exposure to Hepatitis B Virus (HBV), Human Immunodeficiency Virus (HIV) and other bloodborne pathogens that employees may encounter in their workplace.

Respiratory Protection Plan. The respiratory protection plan assures that Accutest employees are protected from exposure to respiratory hazards. This program is used in situations where engineering controls and/or safe work practices do not completely control the identified hazards. In these situations, respirators and other protective equipment are used. Supplemental respiratory protection procedures are applied to specified maintenance personnel, employees who handle hazardous wastes in the hazardous waste storage area, and any employee that voluntarily elects to wear a respirator.

Chemical Hygiene Plan. The Chemical Hygiene Plan complies with the requirements of the Occupational Safety and Health Administration's Occupational Exposure to Hazardous Chemicals in the Laboratory Standard, 29 CFR 1910.1450. This plan establishes procedures, identifies safety equipment, personal protective equipment, and work practices that protect employees from the potential health hazards presented by hazardous chemicals in the laboratory if properly used and/or applied.

Chemical Spill Response Plan. The chemical spill response plan has been designed to minimize the risks from a chemical spill or accidental chemical release in the laboratory. Risk minimization is accomplished through a planned response that follows a defined procedure. The staff has been trained to execute spill response procedures according to the specifications of the plan, which identifies the appropriate action to be taken based on the size of the spill.

Emergency Action & Evacuation Plan. The Emergency Action and Evacuation Plan details the procedures used to protect and safeguard ANC's employees and property during emergencies. Emergencies are defined as fires or explosions, gas leaks, building collapse, hazardous material spills, emergencies that immediately threaten life and health, bomb threats and natural disasters such as floods, hurricanes or tornadoes, terrorism or terrorist actions. The plan identifies and assigns responsibility for executing specific roles in situations requiring emergency action. It also describes the building security actions coinciding with the "Alert Condition", designated by the Department of Homeland Security.

Lockout/Tagout Plan. Lockout/tagout procedures have been established to assure that laboratory employees and outside contractors take steps to render equipment inoperable and/or safe before conducting maintenance activities. The plan details the procedures for conducting maintenance on equipment that has the potential to unexpectedly energize, start up, or release energy or can be operated unexpectedly or accidentally resulting in serious injury to employees. The plan ensures that employees performing maintenance render the equipment safe through lock out or tag out procedures.

Personal Protection Policy. Policies have been implemented which detail the personal protection requirements for employees. The policy includes specifications regarding engineering controls, personal protective equipment (PPE), hazardous waste, chemical exposures, working with chemicals and safe work practices. Safety requirements specific to processes or equipment are reviewed with the department supervisor or the Health and Safety Manager before beginning operations.

Visitor and Contractor Safety Program. A safety brochure is given to all visitors and contractors who visit or conduct business at the facility. The brochure is designed to inform anyone who is not an employee of Accutest Laboratories of the laboratories safety procedures. The brochure directs them to follow all safety programs and plans while on Accutest property. This program also outlines procedures for visitors and contractors in the event of an emergency. Visitors are required to acknowledge receipt and understanding of the Accutest policy annually.

Appendix I

GLOSSARY OF TERMS

Acceptance Criteria: specified limits placed on characteristics of an item, process, or service defined in requirement documents.

Accuracy: the degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components which are due to sampling and analytical operations; a data quality indicator.

Analyst: the designated individual who performs the "hands-on" analytical methods and associated techniques and who is the one responsible for applying required laboratory practices and other pertinent quality controls to meet the required level of quality.

Audit: a systematic evaluation to determine the conformance to quantitative *and qualitative* specifications of some operational function or activity.

Batch: environmental samples that are prepared and/or analyzed together with the same process and personnel, using the same lot(s) of reagents. A preparation batch is composed of one to 20 environmental samples of the same NELAC-defined matrix, meeting the above mentioned criteria and with a maximum time between the start of processing of the first and last sample in the batch to be 24 hours. An analytical batch is composed of prepared environmental samples (extracts, digestates or concentrates) which are analyzed together as a group.

Blank: a sample that has not been exposed to the analyzed sample stream in order to monitor contamination during sampling, transport, storage or analysis. The blank is subjected to the usual analytical and measurement process to establish a zero baseline or background value and is sometimes used to adjust or correct routine analytical results.

Blind Sample: a sub-sample for analysis with a composition known to the submitter. The analyst/laboratory may know the identity of the sample but not its composition. It is used to test the analyst's or laboratory's proficiency in the execution of the measurement process.

Calibration: to determine, by measurement or comparison with a standard, the correct value of each scale reading on a meter, instrument, or other device. The levels of the applied calibration standard should bracket the range of planned or expected sample measurements.

Calibration Curve: the graphical relationship between the known values, such as concentrations of a series of calibration standards and their instrument response.

Calibration Method: a defined technical procedure for performing a calibration.

Calibration Standard: a substance or reference material used to calibrate an instrument. **Certified Reference Material (CRM):** a reference material one or more of whose property values are certified by a technically valid procedure, accompanied by or traceable to a certificate or other documentation, which is issued by a certifying body.

Chain of Custody: an unbroken trail of accountability that ensures the physical security of samples and includes the signatures of all who handle the samples.

Confirmation: verification of the identity of a component through the use of an approach with a different scientific principle from the original method. These may include, but are not limited to second column confirmation, alternate wavelength, derivatization, mass spectral, interpretation, alternative detectors or, additional cleanup procedures.

Corrective Action: the action taken to eliminate the causes of an existing nonconformity, defect or other undesirable situation in order to prevent recurrence.

Data Reduction: the process of transforming raw data by arithmetic or statistical calculations, standard curves, concentration factors, etc., and collation into a more useable form.

Demonstration of Capability: a procedure to establish the ability of the analyst to generate acceptable accuracy.

Document Control: the act of ensuring that documents (and revisions thereto) are proposed, reviewed for accuracy, approved for release by authorized personnel, distributed properly and controlled to ensure use of the correct version at the location where the prescribed activity is performed.

Duplicate Analyses: the analyses or measurements of the variable of interest performed identically on two sub-samples of the same sample. The results from duplicate analyses are used to evaluate analytical or measurement precision but not the precision of sampling, preservation or storage internal to the laboratory.

Field of Testing: NELAC's approach to accrediting laboratories by program, method and analyte. Laboratories requesting accreditation for a program-method-analyte combination or for an updated/improved method are required submit to only that portion of the accreditation process not previously addressed (see NELAC, section 1.9ff).

Laboratory Control Sample (such as laboratory fortified blank, spiked blank, or QC check sample): a sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes from a source independent of the calibration standards or a material containing known and verified amounts of analytes. It is generally used to establish intra-laboratory or analyst specific precision and bias or to assess the performance of all or a portion of the measurement system.

Matrix: the component or substrate that contains the analyte of interest. For purposes of batch and QC requirement determinations, the following matrix distinctions shall be used:

Aqueous: any aqueous sample excluded from the definition of Drinking Water matrix or Saline/Estuarine source. Includes surface water, groundwater, effluents, and TCLP or other extracts.

Drinking Water: any aqueous sample that has been designated a potable or potential potable water source. **Saline/Estuarine:** any aqueous sample from an ocean or estuary, or other salt-water source such as the Great Salt Lake. **Non-aqueous Liquid:** any organic liquid with <15% settleable solids.

Solids: includes soils, sediments, sludges and other matrices with >15% settleable solids.

Chemical Waste: a product or by-product of an industrial process that results in a matrix not previously defined.

Air: whole gas or vapor samples including those contained in flexible or rigid wall containers and the extracted concentrated analytes of interest from a gas or vapor that are collected with a sorbent tube, impinger solution, filter, or other device.

Biota: animal or plant tissue, consisting of entire organisms, homogenates, and/or organ or structure specific subsamples.

Matrix Spike (spiked sample or fortified sample): a sample prepared by adding a known mass of target analyte to a specified amount of matrix sample for which an independent estimate of target analyte concentration is available. Matrix spikes are used, for example, to determine the effect of the matrix on a method's recovery efficiency.

Matrix Spike Duplicate (spiked sample or fortified sample duplicate): a second replicate matrix spike prepared in the laboratory and analyzed to obtain a measure of the precision of the recovery for each analyte.

Method Blank: a sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest, which is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses.

Method Detection Limit: the minimum concentration of a substance (an analyte) that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte.

National Environmental Laboratory Accreditation Program (NELAP): the overall National Environmental Laboratory Accreditation Program.

NELAC Standards: the plan of procedures for consistently evaluating and documenting the ability of laboratories performing environmental measurements to meet nationally defined standards established by the National Environmental Laboratory Accreditation Conference.

Performance Audit: the routine comparison of independently obtained *qualitative and quantitative* measurement system data with routinely obtained data in order to evaluate the proficiency of an analyst or laboratory.

Precision: the degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves; a data quality indicator. Precision is usually expressed as standard deviation, variance or range, in either absolute or relative terms.

Preservation: refrigeration and/or reagents added at the time of sample collection (or later) to maintain the chemical and/or biological integrity of the sample.

Proficiency Testing: a means of evaluating a laboratory's performance under controlled conditions relative to a given set of criteria through analysis of unknown samples provided by an external source.

Proficiency Test Sample (PT): a sample, the composition of which is unknown to the analyst and is provided to test whether the analyst/laboratory can produce analytical results within specified acceptance criteria.

Quality Assurance: an integrated system of activities involving planning, quality control, quality assessment, reporting and quality improvement to ensure that a product or service meets defined standards of quality with a stated level of confidence.

Quality Control: the overall system of technical activities whose purpose is to measure and control the quality of a product or service so that it meets the needs of users.

Quality Manual: a document stating the management policies, objectives, principles, organizational structure and authority, responsibilities, accountability, and implementation of an agency, organization, or laboratory, to ensure the quality of its product and the utility of its product to its users.

Quality System: a structured and documented management system describing the policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an organization for ensuring quality in its work processes, products (items), and services. The quality system provides the framework for planning, implementing, and assessing work performed by the organization and for carrying out required QA and QC.

Reporting Limits: the maximum or minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be quantified with the confidence level required by the data user.

Reagent Blank (method reagent blank or method blank): a sample consisting of reagent(s), without the target analyte or sample matrix, introduced into the analytical procedure at the appropriate point and carried through all subsequent steps to determine the contribution of the reagents and of the involved analytical steps.

Reference Material: a material or substance one or more properties of which are sufficiently well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials.

Reference Method: a method of known and documented accuracy and precision issued by an organization recognized as competent to do so.

Reference Standard: a standard, generally of the highest metrological quality available at a given location, from which measurements made at that location are derived.

Replicate Analyses: the measurements of the variable of interest performed identically on two or more sub-samples of the same sample within a short time interval.

Sample Duplicate: two samples taken from and representative of the same population and carried through all steps of the sampling and analytical procedures in an identical manner. Duplicate samples are used to assess variance of the total method including sampling and analysis.

Spike: a known mass of target analyte added to a blank sample or sub-sample; used to determine recovery efficiency or for other quality control purposes.

Standard: the document describing the elements of laboratory accreditation that has been developed and established within the consensus principles of NELAC and meets the approval requirements of NELAC procedures and policies.

Traceability: the property of a result of a measurement whereby it can be related to appropriate standards, generally international or national standards, through an unbroken chain of comparisons.

Validation: the process of substantiating specified performance criteria.

Work Cell: A defined group of analysts that together perform the method analysis. Members of the group and their specific functions within the work cell must be fully documented. A "work cell" is considered to be all those individuals who see a sample through the complete process of preparation, extraction, or analysis. The entire process is completed by a group of capable individuals; each member of the work cell demonstrates capability for each individual step in the method sequence.

Appendix II : Standard Operational Procedures Directory

Accutest Laboratories Northern California Standard Operating Procedures Directory

GENERAL CHEMISTRY

<u>Number& Revision (-XX)</u>	<u>Standard Operating Procedure Title</u>	<u>Effective Date</u>
GEN001-3	3%Moisture-%Solids CLP ILMO5.3 Exhibit D	05-Jul-10
GEN002-2	Acidity SM2310B	22-Mar-10
GEN003-3	Alkalinity SM2320B	27-Dec-10
GEN004-2	Ammonia SM4500-NH3	16-Jan-10
GEN006-4	Anions EPA 300.0 / SW9056A	04-Jan-11
GEN007-1	Chlorine, Residual SM4500-CL-G / HACH 8167	01-May-09
GEN008-3	Chromium VI Water SM3500-Cr-D / SW7196A	06-Jan-11
GEN009-3	Chromium VI Soil SW3060A / SW7196A	22-Mar-10
GEN010-3	Specific Conductance 120.1 / SM2510B / 9050A	27-Dec-10
GEN011-2	COD Chemical Oxygen Demand SM5220D / HACH 8000	15-Mar-10
GEN012-3	Cyanide, Total and Amenable SM4500-CN-C(Mod),E,G and SW9010C/SW9014	06-Jan-11
GEN013-2	Oil&Grease HEM and SGT-HEM 1664A	13-Feb-10
GEN014-2	Nitrogen, Combined: Nitrate+Nitrite SM4500-E	06-Jan-11
GEN015-2	Nitrogen, Total Kjeldahl TKN SM4500-NH3-F	10-Mar-10
GEN016-2	Nitrogen, Nitrite - NO2 as N SM4500-NO2-B	15-Mar-10
GEN017-3	Fluoride SM4500F-C / SW9214 w/o Distillation	06-Jan-11
GEN018-1	Formaldehyde HACH 8110_Liquid_Not NELAP CMPD	03-Nov-09
GEN019-2	Ferrous Iron SM3500-Fe-D	08-Apr-10
GEN020-1	pH Hydrogen Ion 9040C	08-Apr-10
GEN021-2	Perchlorate by IC Water and Soil 314.0	09-Feb-10
GEN022-2	pH Hydrogen Ion - Soil and Waste 9045C	23-Mar-10
GEN023-2	pH on Water SM4500-H+B	16-Mar-10
GEN024-2	Phenolics, Total Recoverable Soil and Water 420.1 / 9065	17-Mar-10
GEN025-3	Phosphorus, Orthophosphate and Total SM4500-P E	27-Dec-10
GEN026-2	Solids, Settleable SM2540-F	23-Mar-10
GEN027-2	Solids_Total SM18 2540-B	23-Mar-10

GEN028-3	Solids, Total Dissolved SM2540 C	27-Dec-10
GEN029-3	Solids, Total Suspended SM2540_D	27-Dec-10
GEN030-2	Sulfide Hach 8131 - SM4500-S2	17-Mar-10
GEN031-3	TOC Total Organic Carbon SM5310C	06-Jan-11
GEN032-2	Turbidity 180.1 / SM2130B	15-Mar-10
GEN033-0	Water Compatibility ASTM D5058-90C-on line	01-Mar-08
GEN034-2	Glassware Cleaning for Inorganic/Metals	12-Apr-10
GEN035-0	Hexavalent Chromium Water SM21-3500-Cr B	07-Jan-10
GEN036-2	Hexavalent Chromium Water 7196A	24-Mar-10
GEN037-1	Cyanide, Total & Amenable 9010B	16-Mar-10
GEN038-1	Cyanide 9014	16-Mar-10
GEN039-1	Hexavalent Chromium in Water by IC 7199	01-Mar-10
GEN040-1	Hexavalent Chromium in Soil by IC 7199	01-Mar-10
GEN041-0	Oxidation Reduction Potential	18-Nov-09

METALS AND METALS PREP

<u>Number& Revision (-XX)</u>	<u>Standard Operating Procedure Title</u>	<u>Effective Date</u>
MET001-2	Mercury Water 245.1-7470A	10-Mar-10
MET002A-3	Mercury on Solids 7471A	02-Feb-11
MET003-5	Metals by ICP_EPA6010B	08-Oct-10
MET004-3	Metals by ICP_EPA200.7	08-Oct-10
MET005-0	Archiving and Restoring ICP Data	24-Feb-10
MP001-2	Metals Digestions_Aqueous_200.7_Only	26-Feb-10
MP002-2	Metals Digestions_Aqueous_3010A	26-Feb-10
MP003-2	Metals Digestion_Solid_200.7_3050B	11-Feb-10
MP004-0	STLC Waste Extraction Title 22 WET	01-Mar-09

<u>Number&</u>	<u>ORGANIC PREP, VOLATILES AND SEMI VOLATILES Standard Operating Procedure Title</u>	<u>Effective</u>
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<u>Revision (-XX)</u>		<u>Date</u>
OP001-5	Pesticides/PCBs Soil Extraction 3545A	13-Oct-10
OP002-3	Pesticides/PCBs Water Extraction 3510C	10-Dec-09
OP003-1	PCBs Waste Dilution 3580A-on line	04-Dec-08
OP004-3	SVOC Soil Extraction 3545A (ASE) and 3580 (Waste Dilution)	13-Oct-10
OP005-3	SVOC Water Extraction 3510 / 625	16-Feb-10
OP006-3	TEPH Soil Extraction 3545A (ASE)	13-Oct-10
OP007-2	TEPH Water Extraction 3510C	10-Apr-10
OP008-2	Cleaning Procedure for Organic Glassware	23-Feb-10
OP009-2	TCLP_Nonvolatile Analytes (Semi-Vol Organics & Metals)	02-Mar-10
OP010-0	SPLP Synthetic Leaching Procedure for Nonvolatile Analytes	17-Apr-09
OP011-2	NWTPH-HCID & Dx Extraction for Water	05-Mar-10
OP012-3	NWTPH-HCID & Dx Extraction for Soil	01-Nov-10
OP013-1	TCLP for Volatiles ZHE-on line	30-Apr-10
OP016-0	Solid Extraction for Total Petroleum Hydrocarbon (TPH)	28-Apr-10
	SW846 Method 3550C_Ultrasonic Extraction	
OP017-0	Solid Extraction for Hydrocarbon Identification_NWTPH-HCID and NWTPH-Dx Method Using Sonicator	28-Apr-10
SV001-7	Pesticide / PCB Analysis by 8081A / 8082A	01-Nov-10
SV002-3	SVOC Analysis by 8270C	17-Dec-09
SV003-2	TPH-Extractable Analysis by 8015B	15-May-10
SV004-1	NWTPH-HCID	08-Mar-10
SV005-2	NWTPH-Dx	07-Feb-11
SV006-3	Analysis of TPH-Extractable by EPA 8015B_8015AZ GC/FID	23-Dec-10
SV007-0	Base_Neutral_Acid_EPA_625	27-Jan-10
SV008-0	Analysis of Organochlorine Pesticides and Polychlorinated Biphenyls	14-Mar-10
VO001-5	Volatile Organic Compounds by GC/MS 8260B	04-Jan-11
VO001T1-0	DoD QSMv4.1 8260 Table F-4	01-Jan-10
VO002-2	BTEX / MtBE / TPH as Gasoline by 8021/8015BM	07-Jan-10
VO003-1	Alcohols (MeOH/EtOH) by 8015 Aqueous Injection	12-Feb-09
VO004-1	Volatile Organics - Refrigerator Blanks-on line	28-Oct-09
VO005-1	Purge & Trap Soils 5035	25-May-10
VO006-0	Purge & Trap Waters 5030	16-Feb-09
VO008-1	Volatile Organic Compound by EPA 624 (CWA)	06-Dec-10
VO009-0	NWTPH-Gx and VOCs by GC/MS	29-May-09
VO010-1	NWTPH-Gx by GC-FID	31-Jan-11

PROJECT MANAGEMENT

<u>Number& Revision (-XX)</u>	<u>Standard Operating Procedure Title</u>	<u>Effective Date</u>
PM001-1	Project Management	19-Oct-09
PM002-1	Review of Tenders	19-Oct-09
PM003-2	Subcontracting	04-Jan-10
PM003A-1	Subcontract Reporting Procedure	15-Oct-09
PM004-1	Confidentiality Protection Procedures	09-Oct-09

QUALITY ASSURANCE

<u>Number& Revision (-XX)</u>	<u>Standard Operating Procedure Title</u>	<u>Effective Date</u>
QA001-2	Error Correction	06-Jul-10
QA002-2	Corrective Action	12-Feb-10
QA003-0	Blank Spike Control Charting-on line	01-Mar-08
QA004-2	Measurement Traceability-Purchasing Services and Laboratory Supplies	11-Feb-10
QA005-1	Auto Pipette Calibration	25-Jan-09
QA006-2	MDL Determination	07-Apr-10
QA008-8	Quality System Manual for Accutest Northern California	21-Feb-11
QA009-2	Refrigerator and Freezer Temperatures	06-Feb-10
QA010-3	Annual Verification of Thermometer Accuracy	29-Nov-10
QA011-2	Test Method Validation MDL, LOD, LOQ	02-Feb-11
QA012-1	Significant Figures	02-Apr-09
QA013-2	Manual Integrations	17-Apr-09
QA014-2	Personnel Training Documentation _DOC	12-Feb-10
QA015-1	Signature Implies Approval	28-Sept-10
QA015-1a	Signature Authority	02-Feb-11
QA016-2	SOP Template	09-Feb-10
QA017-1	Data Integrity Investigations	09-Oct-09
QA018-1	Sample Batching for Prep and Analysis	15-Oct-09
QA019-1	Support Equipment and Maintenance	27-Oct-09
QA020-2	SOPs: Preparation, Approval, Distribution and Archiving	12-Feb-10
QA021-1	Control of Laboratory Documentation	12-Oct-09
QA022-1	Quality Control, Evaluation Criteria (ME and Bias)	27-Oct-09
QA023-1	Sample Compositing and Sample Aliquots	29-Oct-09
QA024-1	PT Proficiency Testing	03-Apr-09
QA025-1	ReportGeneration_DataPackageReview	05-Oct-09
QA026-0	Sample Homogenization (Representative Solid Sample Aliquots)	13-Jan-09
QA027-0	Syringe Calibration	17-Apr-09
QA028-0	Volumetric Dispensers - Critical Volumes	20-Jan-09
QA029-1	Volumetric Dispensers - Non-Critical Volumes	08-Jul-10
QA030-0	Calibration Check of Analytical Balances	17-Feb-09
QA031-0	Data Integrity and Ethics Training	19-Oct-09
QA032-0	Review of Inorganic Data	26-Oct-09

QA033-1	Control of Non-Conforming Work	21-Feb-11
QA034-0	Quality System Review	07-Sep-09
QA035-1	Internal Audit and Preventive Action	12-Feb-10
QA036-0	Data Integrity Monitoring	23-Oct-09
QA037-0	Data Integrity Issues Reporting	20-Oct-09
QA038-0	Client Complaints Resolution	19-Oct-09
QA039-0	Control of Records	20-Oct-09
QA040-0	In-house QC Criteria - Development and Use	26-Oct-09
QA041-0	Modified Method Comparability and Verification	26-Oct-09
QA042-0	Review of Organic Data	25-Oct-09
QA043-0	Purchasing Services and Laboratory Supplies	10-Feb-10
QA044-0	DI Water Quality Control Procedure	24-Feb-10

SAMPLE CONTROL

<u>Number& Revision (-XX)</u>	<u>Standard Operating Procedure Title</u>	<u>Effective Date</u>
SC001-2	Sample Handling and Login Procedures	14-Jan-09
SC002-2	Waste (including Samples) Disposal	05-Jan-09
SC003-0	Daily Thermometer Temperature Record-on line	25-Feb-08
SC004-1	Sample and Analysis Guide	01-Jan-09
SC005-1	Sample Storage	23-Oct-09
SC006-0	Sample Container Receipt and Quality Control –on line	05-Feb-10
SC008-0	Foreign Soil Handling	23-Dec-09

ADMINISTRATION

<u>Number& Revision (-XX)</u>	<u>Standard Operating Procedure Title</u>	<u>Effective Date</u>
ADM001-1	Procedure for Conducting Management Reviews	05-Oct-09
ADM002-1	Data Security & Integrity Procedure	20-Oct-09
ADM003-1	Job Descriptions	06-Oct-09
ADM004-0	Procedure for Requesting and Verifying Software	26-Oct-09

For the most current listing, go to:
 \\Accunca.accutest.com\depts\qa\sops\sop_completelist_2010\current_active_sop_oct_2010

Appendix III: Analytical Capability**NELAP No EPA Method****102 - Inorganic Chemistry of Drinking Water**

102.030.003	EPA 300.0
102.045.001	EPA 314.0
102.220.001	SM4500-NO2 B
102.240.001	SM4500-P E

108 - Inorganic Chemistry of Wastewater

108.020.001	EPA 120.1
108.381.001	EPA 1664A
108.110.001	EPA 180.1
108.112.001	EPA 200.7
108.120.001	EPA 300.0
108.360.001	EPA 420.1
108.660.001	HACH8000
108.390.001	SM2130B
108.400.001	SM2310B
108.410.001	SM2320B
108.420.001	SM2340B
108.430.001	SM2510B
108.440.001	SM2540B
108.441.001	SM2540C
108.442.001	SM2540D
108.443.001	SM2540F
108.465.001	SM4500-CI G
108.470.001	SM4500-CN C
108.472.001	SM4500-CN E
108.473.001	SM4500-CN G
108.490.001	SM4500-H+ B
108.493.001	SM4500-NH3 D or E (19th/20th)
108.495.001	SM4500-NH3 E (18th)
108.494.001	SM4500-NH3 F or G (18th)
108.510.001	SM4500-NO2 B
108.520.001	SM4500-NO3 E
108.540.001	SM4500-P E
108.580.001	SM4500-S= D
108.611.001	SM5310C

109 - Toxic Chemical Elements of Wastewater

109.010.001	EPA 200.7
109.190.001	EPA 245.1
109.808.001	SM3500-Cr B (21st)
109.811.001	SM3500-Cr D (18th/19th)
109.825.001	SM3500-Fe D (18th/19th)

110 - Volatile Organic Chemistry of Wastewater

110.040.001	EPA 624
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111 - Semi-volatile Organic Chemistry of Wastewater

111.273.001	EPA 1664A
111.170.001	EPA 608
111.100.001	EPA 625
111.272.001	SM5520B (20th)

114 - Inorganic Chemistry of Hazardous Waste

114.010.001	EPA 6010B
114.103.001	EPA 7196A
114.106.001	EPA 7199
114.140.001	EPA 7470A
114.141.001	EPA 7471A
114.222.001	EPA 9014
114.240.001	EPA 9040B
114.241.001	EPA 9045C
114.250.001	EPA 9056
114.270.001	EPA 9214

115 - Extraction Test of Hazardous Waste

115.030.001	CCR Chapter11, Article 5, Appendix II
115.021.001	EPA 1311
115.040.001	EPA 1312

116 - Volatile Organic Chemistry of Hazardous Waste

116.020.009	EPA 8015B
116.040.002	EPA 8021B
116.080.001	EPA 8260B
116.110.001	LUFT
116.100.001	LUFT GC/MS

117 - Semi-volatile Organic Chemistry of Hazardous Waste

117.010.001	EPA 8015B
117.210.000	EPA 8081A
117.220.000	EPA 8082
117.110.000	EPA 8270C
117.016.001	LUFT

120 - Physical Properties of Hazardous Waste

120.070.001	EPA 9040B
120.080.001	EPA 9045C

For the most current listing, go to:
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Appendix IV: Equipment List

Equip.	Manufacture& Description	Serial Number	Operating System Software	Data Processing Software	Location
GC-JJ	Hewlett-Packard 5890/PID-FID/LSC2000/G1530A	US00025879	HP Chemstation	HP Chemstation	Organics Volatiles
GC-KK	Hewlett-Packard 5890/PID-FID/7673/18593B/18594B/ G1205A/	3235A46302	HP Chemstation	HP Chemstation	Organics Volatiles
GCMS-L	Agilent Technologies 6890/5975C/4660A	CN10706098	HP Chemstation	HP Chemstation	Organics Volatiles
GCMS-M	Hewlett-Packard 6890/5973N MS Tekmar LSC 2000/G1530A/G1098A	US00021444	HP Chemstation	HP Chemstation	Organics Volatiles
GCMS-N	Hewlett-Packard 6890/5973N MS Tekmar LSC 3000/ G1530 A	US00027818	HP Chemstation	HP Chemstation	Organics Volatiles
GCMS-V	Agilent Technologies 6890N/5973N/ Tekmar LSC 2000/G2577A/G1098A	US10518041	HP Chemstation	HP Chemstation	Organics Volatiles
GCMS-R	Agilent Technologies 7890A/5975C O.I. Analytical/4551-A	US10452710	HP Chemstation	HP Chemstation	Organics Volatiles
GCMS-Q	Agilent Technologies 7890A/5975C O.I. Analytical/4551-A	US10452714	HP Chemstation	HP Chemstation	Organics Volatiles
GCMS-W	Agilent Technologies 6890/5975B G3172A/4552/OI 4660	CN10627031	HP Chemstation	HP Chemstation	Organics Volatiles
GC-FF	Hewlett-Packard 5890/ FID/7673 18593A/18596A/18594A	3140A38168	HP Chemstation	HP Chemstation	Organics SVOCs
GC-GG	Hewlett-Packard 6890/ FID/7673 18593B/18596M/G1512A	US00001432	HP Chemstation	HP Chemstation	Organics SVOCs
GC-HH	Hewlett-Packard 6890/ FID/7673 18593A/18596B/G1701BA	3310A48782	HP Chemstation	HP Chemstation	Organics SVOCs
GC-OO	Hewlett-Packard 5890/ECD/7673 G1223A/18593B/18594B/18596B	3235A44943	HP Chemstation	HP Chemstation	Organics SVOCs
GC-PP	Hewlett-Packard 5890/ECD/7673A G1223A/18593B/18594B/18596B	3115A34621	HP Chemstation	HP Chemstation	Organics SVOCs
GCMS-X	Agilent Technologies 6890N/5975B/7683 G3172A/G2913A/G2614A	CN10636038	HP Chemstation	HP Chemstation	Organics SVOCs
GCMS-Y	Agilent Technologies 6890N/5975B/7683 G3172A/G2613A/G2614A/G1701DA	CN10634077	HP Chemstation	HP Chemstation	Organics SVOCs
ASE-2	Dionex (Accelerated Solvent Extractor)	07020400	None	None	Organic Prep

Equip.	Manufacture& Description	Serial Number	Operating System Software	Data Processing Software	Location
ASE-3	Dionex (Accelerated Solvent Extractor)	07030593	None	None	Organic Prep
LER-1	Tumbler	Bison 508-01-53	None	None	Organic Prep
TUMBLER-2	Tumbler	GTR Tumbler	None	None	Organic Prep
TUMBLER-3	Tumbler	GTR Tumbler	None	None	Organic Prep
Centrifuge	Fisher Scientific	225	None	None	Organic Prep
TV1	Concentrator/ TurboVap LV	TV0626N13155	None	None	Organic Prep
TV2	Concentrator/ TurboVap LV	TV0553N12782	None	None	Organic Prep
TV3	Concentrator/ TurboVap LV	TV9907N18679	None	None	Organic Prep
TV4	Concentrator/ TurboVap LV	TV0105N10106	None	None	Organic Prep
Sonicator Bath	Aquasonic 50T	21811-820	None	None	Organic Prep
Sonicator	Misomix Ultrasonic Liquid Processor	-	None	None	Organic Prep
Balance	Adventurer Pro AV8101C	8027331004	None	None	Organic Prep
Oven	Oven	1330GZZZMFG	None	None	Organic Prep
Shaker-1	Shaker	3520	None	None	Organic Prep
Shaker-2	Shaker	3520	None	None	Organic Prep
Shaker-3	Shaker	SHKA 2506	None	None	Organic Prep

Equip.	Manufacture& Description	Serial Number	Operating System Software	Data Processing Software	Location
Shaker-4	Shaker	SHKA 2506	None	None	Organic Prep
VORTEX-	Vortex	Genie2	None	None	Organic Prep
VORTEX-	Vortex	Genie2	None	None	Organic Prep
Mercury U	Mercury Amalgam Unit	Amalgam System	None	None	Mercury
BLOCK-1	Block Digester-ICP	SC100 SCP Digiprep 24	None	None	Metals Prep
BLOCK-2	Block Digester-ICP	SC100 SCP Digiprep 24	None	None	Metals Prep
BLOCK-3	Block Digester-ICP	SC100 SCP Digiprep 36	None	None	Metals Prep
BLOCK-4	Block Digester-Hg	05-C0530 Digiprep 24	None	None	Metals Prep
FIMS-1	Mercury Analyzer/ FIMS-100	1522	None	None	Metals Prep
ICP-1	Perkin Elmer ICP Chiller, OES-Dual View, AS93/ 5300DV	077N4112001	None	None	Metals
iCAP6000 Series	Thermo Fisher Scientific	ICP-20104815	None	None	Metals
MET-1	Balance XL-500	SN08675	None	None	Metals Prep
IC-1	Dionex IC25/AS40/AS12/AS12G	01100231 02010170	Chromeleon	Chromeleon	Inorganics
IC-2	Dionex ICS2000/AS40/AS16G	04030440 09010526	Chromeleon	Chromeleon	Inorganics
IC-3	Dionex ICS3000/ASDV/NG1	08050056 09101340 07080459	Chromeleon	Chromeleon	Inorganics
Meter-1	Meter, ISE	090756	None	None	Inorganics
Meter-2	Meter, ISE	008802	None	None	Inorganics
Meter-3	pH Meter	001103	None	None	Inorganics

Equip.	Manufacture& Description	Serial Number	Operating System Software	Data Processing Software	Location
Meter-4	Meter, ISE	B19733	None	None	Inorganics
Vacuum P	Vacuum Pump	DOA-P704-AA	None	None	Inorganics
Chiller	1170	305421	None	None	Inorganics
Chiller	1171MD	F09900116	None	None	Inorganics
Meter	Conductivity 162A	017809	None	None	Inorganics
COD	COD Reactor 16500-10	2329	None	None	Inorganics
Vacuum P	Vacuum Pump	DOA-P704-AA	None	None	Inorganics
Vacuum P	Vacuum Pump	400-1901	None	None	Inorganics
SPE	Manifold-6 position	-	None	None	Inorganics
Centrifuge	Sorvall Legend XT	40970026	None	None	Inorganics
Hot Plate	PC-600D	013606254903	None	None	Inorganics
Hot Plate	Hot Plate	980061119531	None	None	Inorganics
NTU-1	Nephelometer 2100AN	05090C020055	None	None	Inorganics
OVEN-1	Oven 1350GM	0200403	None	None	Inorganics
OVEN-2	Oven OV702G	2039090215821	None	None	Inorganics
SPEC-1	Spectrophotometer DR/2500 Odyssey	011000001384	None	None	Inorganics
SPEC-2	Spectrophotometer Genesys 20	35GL317012	None	None	Inorganics
TOC	TOC Autosampler/Analyzer Phoenix 8000	US06209002	None	None	Inorganics
WCL-1	Balance AG204	1119362894	None	None	Inorganics
WCL-2	Balance BA-610	20701774	None	None	Inorganics

Attachment C
USEPA Method TO-10A

**Compendium of Methods
for the Determination of
Toxic Organic Compounds
in Ambient Air**

Second Edition

Compendium Method TO-10A

**Determination Of Pesticides And
Polychlorinated Biphenyls In Ambient
Air Using Low Volume Polyurethane
Foam (PUF) Sampling Followed By
Gas Chromatographic/Multi-Detector
Detection (GC/MD)**

**Center for Environmental Research Information
Office of Research and Development
U.S. Environmental Protection Agency
Cincinnati, OH 45268**

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Method TO-10A

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DISCLAIMER

This Compendium has been subjected to the Agency's peer and administrative review, and it has been approved for publication as an EPA document. Mention of trade names or commercial products does not constitute endorsement or recommendation for use.

METHOD TO-10A

Determination Of Pesticides And Polychlorinated Biphenyls In Ambient Air Using Low Volume Polyurethane Foam (PUF) Sampling Followed By Gas Chromatographic/Multi-Detector Detection (GC/MD)

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METHOD TO-10A

Determination Of Pesticides And Polychlorinated Biphenyls In Ambient Air Using Low Volume Polyurethane Foam (PUF) Sampling Followed By Gas Chromatographic/Multi-Detector (GC/MD) Detection

1. Scope

1.1 This document describes a method for sampling and analysis of a variety of common pesticides and for polychlorinated biphenyls (PCBs) in ambient air. The procedure is based on the adsorption of chemicals from ambient air on polyurethane foam (PUF) or a combination of PUF and granular sorbent using a low volume sampler.

1.2 The low volume PUF sampling procedure is applicable to multicomponent atmospheres containing common pesticide concentrations from 0.001 to 50 $\mu\text{g}/\text{m}^3$ over 4- to 24-hour sampling periods. The limits of detection will depend on the nature of the analyte and the length of the sampling period.

1.3 Specific compounds for which the method has been employed are listed in Table 1. The analytical methodology described in Compendium Method TO-10A is currently employed by laboratories throughout the U.S. The sampling methodology has been formulated to meet the needs of common pesticide and PCB sampling in ambient air.

1.4 Compendium Method TO-10 was originally published in 1989. The method was further modified for indoor air application in 1990. In an effort to keep the method consistent with current technology, Compendium Method TO-10 has incorporated ASTM Method D4861-94 (1) and is published here as Compendium Method TO-10A.

2. Summary of Method

2.1 A low-volume (1 to 5 L/minute) sample is used to collect vapors on a sorbent cartridge containing PUF or PUF in combination with another solid sorbent. Airborne particles may also be collected, but the sampling efficiency is not known (2).

2.2 Pesticides and other chemicals are extracted from the sorbent cartridge with 5 percent diethyl ether in hexane and determined by gas chromatography coupled with an electron capture detector (ECD), nitrogen-phosphorus detector (NPD), flame photometric detector (FPD), Hall electrolytic conductivity detector (HECD), or a mass spectrometer (MS). For common pesticides, high performance liquid chromatography (HPLC) coupled with an ultraviolet (UV) detector or electrochemical detector may be preferable. This method describes the use of an electron capture detector.

2.3 Interferences resulting from analytes having similar retention times during GC analysis are resolved by improving the resolution or separation, such as by changing the chromatographic column or operating parameters, or by fractionating the sample by column chromatography.

3. Significance

3.1 Pesticide usage and environmental distribution are common to rural and urban areas of the United States. The application of pesticides can cause potential adverse health effects to humans by contaminating soil, water, air, plants, and animal life. However, human exposure to PCBs continues to be a problem because of their presence in the environment.

3.2 Many pesticides and PCBs exhibit bioaccumulative, chronic health effects; therefore, monitoring the presence of these compounds in ambient air is of great importance.

3.3 Use of a portable, low volume PUF sampling system allows the user flexibility in locating the apparatus. The user can place the apparatus in a stationary or mobile location. The portable sampling apparatus may be positioned in a vertical or horizontal stationary location (if necessary, accompanied with supporting structure). Mobile positioning of the system can be accomplished by attaching the apparatus to a person to test air in the individual's breathing zone.

3.4 Moreover, this method has been successfully applied to measurement of common pesticides in outdoor air, indoor air and for personal respiratory exposure monitoring (3).

4. Applicable Documents

4.1 ASTM Standards

- D1356 *Definition of Terms Relating to Atmospheric Sampling and Analysis*
- D4861-94 *Standard Practice for Sampling and Analysis of Pesticides and Polychlorinated Biphenyls in Air*
- E260 *Recommended Practice for General Gas Chromatography Procedures*
- E355 *Practice for Gas Chromatography Terms and Relationships*
- D3686 *Practice for Sampling Atmospheres to Collect Organic Compound Vapors (Activated Charcoal Tube Adsorption Method)*
- D3687 *Practice for Analysis of Organic Compound Vapors Collected by the Activated Charcoal Tube Adsorption*
- D4185 *Practice for Measurement of Metals in Workplace Atmosphere by Atomic Absorption Spectrophotometry*

4.2 EPA Documents

- *Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air: Method TO-10, Second Supplement*, U. S. Environmental Protection Agency, EPA 600/4-89-018, March 1989.
- *Manual of Analytical Methods for Determination of Pesticides in Humans and Environmental Standards*, U. S. Environmental Protection Agency, EPA 600/8-80-038, June 1980.
- *Compendium of Methods for the Determination of Air Pollutants in Indoor Air: Method IP-8*, U. S. Environmental Protection Agency, EPA 600/4-90-010, May 1990.

4.3 Other Documents

- Code of Federal Regulations, Title 40, Part 136, Method 604

5. Definitions

[Note: Definitions used in this document and in any user-prepared Standard operating procedures (SOPs) should be consistent with ASTM D1356, E260, and E355. All abbreviations and symbols are defined within this document at point of use.]

5.1 Sampling efficiency (SE)-ability of the sampling medium to trap analytes of interest. The percentage of the analyte of interest collected and retained by the sampling medium when it is introduced as a vapor in air or nitrogen into the air sampler and the sampler is operated under normal conditions for a period of time equal to or greater than that required for the intended use is indicated by %SE.

5.2 Retention efficiency (RE)-ability of sampling medium to retain a compound added (spiked) to it in liquid solution.

5.3 Static retention efficiency-ability of the sampling medium to retain the solution spike when the sample cartridge is stored under clean, quiescent conditions for the duration of the test period.

5.4 Dynamic retention efficiency (RE_d)-ability of the sampling medium to retain the solution spike when air or nitrogen is drawn through the sampling cartridge under normal operating conditions for the duration of the test period. The dynamic RE is normally equal to or less than the SE.

5.5 Retention time (RT)-time to elute a specific chemical from a chromatographic column, for a specific carrier gas flow rate, measured from the time the chemical is injected into the gas stream until it appears at the detector.

5.6 Relative retention time (RRT)-a ratio of RTs for two chemicals for the same chromatographic column and carrier gas flow rate, where the denominator represents a reference chemical.

5.7 Surrogate standard-a chemically inert compound (not expected to occur in the environmental sample) that is added to each sample, blank, and matrix-spiked sample before extraction and analysis. The recovery of the surrogate standard is used to monitor unusual matrix effects, gross sample processing errors, etc. Surrogate recovery is evaluated for acceptance by determining whether the measured concentration falls within acceptable limits.

6. Interferences

6.1 Any gas or liquid chromatographic separation of complex mixtures of organic chemicals is subject to serious interference problems due to coelution of two or more compounds. The use of capillary or microbore columns with superior resolution or two or more columns of different polarity will frequently eliminate these problems. In addition, selectivity may be further enhanced by use of a MS operated in the selected ion monitoring (SIM) mode as the GC detector. In this mode, co-eluting compounds can often be determined.

6.2 The ECD responds to a wide variety of organic compounds. It is likely that such compounds will be encountered as interferences during GC/ECD analysis. The NPD, FPD, and HECD detectors are element specific, but are still subject to interferences. UV detectors for HPLC are nearly universal, and the electrochemical detector may also respond to a variety of chemicals. Mass spectrometric analyses will generally provide positive identification of specific compounds.

6.3 PCBs and certain organochlorine pesticides (e.g., chlordane) are complex mixtures of individual compounds which can cause difficulty in accurately quantifying a particular formulation in a multiple component mixture. PCBs may interfere with the determination of pesticides.

6.4 Contamination of glassware and sampling apparatus with traces of pesticides or PCBs can be a major source of error, particularly at lower analyte concentrations. Careful attention to cleaning and handling procedures is required during all steps of sampling and analysis to minimize this source of error.

6.5 The general approaches listed below should be followed to minimize interferences.

6.5.1 Polar compounds, including certain pesticides (e.g., organophosphorus and carbamate classes) can be removed by column chromatography on alumina. Alumina clean-up will permit analysis of most organochlorine pesticides and PCBs (4).

6.5.2 PCBs may be separated from other organochlorine pesticides by column chromatography on silicic acid (5,6).

6.5.3 Many pesticides can be fractionated into groups by column chromatography on Florisil (6).

7. Equipment and Materials

7.1 Materials for Sample Collection

7.1.1 Continuous-Flow Sampling Pump (see Figure 1). The pump should provide a constant air flow ($\leq \pm 5\%$), be quiet and unobtrusive, with a flow rate of 1 to 5 L/min. Sources of equipment are Supelco, Supelco Park, Bellefonte, PA; SKC, 334 Valley View Road, Eighty Four, PA and other manufacturers.

7.1.2 Sampling Cartridge (see Figure 2). Constructed from a 20-mm (I.D.) x 10-cm borosilicate glass tube drawn down to a 7-mm (O.D.) open connection for attachment to the pump by way of flexible tubing (see Figure 1).

7.1.3 Sorbent, Polyurethane Foam (PUF). Cut into a cylinder, 22-mm I.D. and 7.6-cm long, fitted under slight compression inside the cartridge. The PUF should be of the polyether type, (density of 0.0225 g/cm³). This is the type of foam used for furniture upholstery, pillows, and mattresses. The PUF cylinders (plugs) should be slightly larger in diameter than the internal diameter of the cartridge. The PUF sorbent may be cut by one of the following means:

- With a high-speed cutting tool, such as a motorized cork borer. Distilled water should be used to lubricate the cutting tool.
- With a hot wire cutter. Care should be exercised to prevent thermal degradation of the foam.
- With scissors, while plugs are compressed between the 22-mm circular templates.

Alternatively, pre-extracted PUF plugs and glass cartridges may be obtained commercially.

7.1.4 Particle Filter. The collection efficiency of PUF for small-diameter (0.1 to 1 μm) airborne particles is only about 20% (7). However, most pesticides and PCBs exist in air under steady-state conditions primarily as vapors (8). Most particulate-associated pesticides or PCBs, if any, will also tend to be vaporized from filters after collection (9). Collocated sampling with and without a quartz-fiber pre-filter has yielded indistinguishable results for a broad spectrum of pesticides and PCBs found in indoor air (10).

7.1.4.1 An open-face filter may be attached to the sampling cartridge by means of a union for 1-in. (25.4-mm) tubing.

7.1.4.2 A 32-mm diameter quartz microfiber filter (e.g., Palifelex® type 2500 QAT-UP) is placed in the open end of the union and supported by means of a screen or perforated metal plate [e.g., a 304-stainless steel disk, 0.0312-in. (0.8-mm) thick with 1/16-in. (1.6-mm) diameter round perforations at 132 holes per in.² (20 holes/cm²), 41% open area.]. A 32-mm Viton® O-ring is placed between the filter and outer nut to effect a seal (see Figure 3). This filter holder is available from Supelco Park, Bellefonte, PA; SKC, 334 Forty Eight, PA; and other manufacturers.

7.1.5 Size-Selective Impactor Inlet. A size-selective impactor inlet with an average particle-size cut-point of 2.5 μm or 10 μm mean diameter at a sampling rate of 4 L/min may be used to exclude nonrespirable airborne particulate matter (11). This inlet, particle filter support, sampling cartridge holders are available commercially from Supelco, Supelco Park, Bellefonte, PA; SKC, 334 Forty Eight, PA and University Research Glassware (URG), Chapel Hill, NC.

7.1.6 Tenax-TA. 60/80 mesh, 2,6-diphenylphenylene oxide polymer. Commercially available from Supelco, Supelco Park, Bellefonte, PA and SKC, 334 Forty Eight, PA.

7.2 Equipment for Analysis

7.2.1 Gas Chromatograph (GC). The GC system should be equipped with appropriate detector(s) and either an isothermally controlled or temperature programmed heating oven. Improved detection limits may be obtained with a GC equipped with a cool on-column or splitless injector.

7.2.2 Gas Chromatographic Column. As an example, a 0.32 mm (I.D.) x 30 m DB-5, DB-17, DB-608, and DB-1701 are available. Other columns may also provide acceptable results.

7.2.3 HPLC Column. As an example, a 4.6-mm x 25-cm Zorbax SIL or μ Bondpak C-18. Other columns may also provide acceptable results.

7.2.4 Microsyringes. 5 μL volume or other appropriate sizes.

7.3 Reagents and Other Materials

7.3.1 Round Bottom Flasks. 500 mL, T 24/40 joints, best source.

7.3.2 Capacity Soxhlet Extractors. 300 mL, with reflux condensers, best source.

7.3.3 Kuderna-Danish Concentrator. 500 mL, with Snyder columns, best source.

7.3.4 Graduated Concentrator Tubes. 10 mL, with 19/22 stoppers, best source.

7.3.5 Graduated Concentrator Tubes. 1 mL, with 14/20 stoppers, best source.

7.3.6 TFE Fluorocarbon Tape. 1/2 in., best source.

7.3.7 Filter Tubes. Size 40 mm (I.D.) x 80 mm.

7.3.8 Serum Vials. 1 mL and 5 mL, fitted with caps lined with TFE fluorocarbon.

7.3.9 Pasteur Pipettes. 9 in., best source.

7.3.10 Glass Wool. Fired at 500°C, best source.

7.3.11 Boiling Chips. Fired at 500°C, best source.

7.3.12 Forceps. Stainless steel, 12 in., best source.

7.3.13 Gloves. Latex or precleaned (5% ether/hexane Soxhlet extracted) cotton.

- 7.3.14 Steam Bath.
- 7.3.15 Heating Mantles. 500 mL.
- 7.3.16 Analytical Evaporator. Nitrogen blow-down.
- 7.3.17 Acetone. Pesticide quality.
- 7.3.18 n-Hexane. Pesticide quality.
- 7.3.19 Diethyl Ether. Preserved with 2% ethanol.
- 7.3.20 Sodium Sulfate. Anhydrous analytical grade.
- 7.3.21 Alumina. Activity Grade IV, 100/200 mesh.
- 7.3.22 Glass Chromatographic Column. 2-mm I.D. x 15-cm long.
- 7.3.23 Soxhlet Extraction System. Including Soxhlet extractors (500 and 300 mL), variable voltage transformers, and cooling water source.
- 7.3.24 Vacuum Oven. Connected to water aspirator.
- 7.3.25 Die.
- 7.3.26 Ice Chest.
- 7.3.27 Silicic Acid. Pesticide grade.
- 7.3.28 Octachloronaphthalene (OCN). Research grade.
- 7.3.29 Florisil. Pesticide grade.

8. Assembly and Calibration of Sampling System

8.1 Description of Sampling Apparatus

8.1.1 A typical sampling arrangement utilizing a personal air pump is shown in Figure 1. This method is designed to use air sampling pumps capable of pulling air through the sampling cartridge at flow rates of 1 to 5 L/min. The method writeup presents the use of this device.

8.1.2 The sampling cartridge (see Figure 2) consists of a glass sampling cartridge in which the PUF plug or PUF/Tenax® TA "sandwich" is retained.

8.2 Calibration of Sampling System

8.2.1 Air flow through the sampling system is calibrated by the assembly shown in Figure 4. All air sampler must be calibrated in the laboratory before and after each sample collection period, using the procedure described below.

8.2.2 For accurate calibration, attach the sampling cartridge in-line during calibration. Vinyl bubble tubing or other means (e.g., rubber stopper or glass joint) may be used to connect the large end of the cartridge to the calibration system. Refer to ASTM Practice D3686 or D4185, for procedures to calibrate small volume air pumps.

9. Preparation of PUF Sampling Cartridges

9.1 The PUF adsorbent is white and yellows upon exposure to light. The "yellowing" of PUF will not affect its ability to collect pesticides or PCBs.

9.2 For initial cleanup and quality assurance purposes, the PUF plug is placed in a Soxhlet extractor and extracted with acetone for 14 to 24 hours at 4 to 6 cycles per hour.

[Note: If commercially pre-extracted PUF plugs are used, extraction with acetone is not required.]

Follow with a 16-hour Soxhlet extraction with 5% diethyl ether in n-hexane. When cartridges are reused, 5% diethyl ether in n-hexane can be used as the cleanup solvent.

9.3 Place the extracted PUF in a vacuum oven connected to a water aspirator and dry at room temperature for 2 to 4 hours (until no solvent odor is detected). Alternatively, they may be dried at room temperature in an air-tight container with circulating nitrogen (zero grade). Place the clean PUF plug into a labeled glass sampling cartridges using gloves and forceps. Wrap the cartridges with hexane-rinsed aluminum foil and placed in jars fitted with TFE fluorocarbon-lined caps. The foil wrapping may also be marked for identification using a blunt probe.

9.4 Granular sorbents may be combined with PUF to extend the range of use to compounds with saturation vapor pressures greater than 10^{-4} kPa (6). A useful combination trap can be assembled by "sandwiching" 0.6 g of Tenax-TA between two 22-mm I.D. x 3.8-cm pre-cleaned PUF plugs, as shown in Figure 2, Cartridge b. The Tenax-TA should be pre-extracted as described in Section 9.2. This trap may be extracted, vacuum dried, and removed without unloading it.

9.5 Analyze at least one assembled cartridge from each batch as a laboratory blank before the batch is acceptable. A blank level of <10 ng/plug for single component compounds is considered to be acceptable. For multiple component mixtures (e.g., PCBs), the blank level should be <100 ng/plug.

9.6 After cleaning, cartridges are considered clean up to 30 days when stored in sealed containers. Certified clean cartridges do not need to be chilled when shipping to the field.

10. Sampling

[Note: After the sampling system has been assembled and calibrated as per Section 8, it can be used to collect air samples as described below. The prepared sample cartridges should be used within 30 days of certification and should be handled only with latex or precleaned cotton gloves.]

10.1 Carefully remove the clean sample cartridge from the aluminum foil wrapping (the foil is returned to jars for later use) and attached to the pump with flexible tubing. The sampling assembly is positioned with the intake downward or in horizontal position. Locate the sampler in an unobstructed area at least 30 meters from any obstacle to air flow. The PUF or PUF/XAD-2 cartridge intake is positioned 1 to 2 m above ground level. Cartridge height above ground is recorded on the Compendium Method TO-10A field test data sheet (FTDS), as illustrated in Figure 5.

10.2 After the PUF cartridge is correctly inserted and positioned, the power switch is turned on and the sampling begins. The elapsed time meter is activated and the start time is recorded. The pumps are checked during the sampling process and any abnormal conditions discovered are recorded on the FTDS. Ambient temperatures and barometric pressures are measured and recorded periodically during the sampling procedure on the FTDS.

10.3 At the end of the desired sampling period, the power is turned off, the PUF cartridge removed from the sampler and wrapped with the original aluminum foil and placed in a sealed, labeled container for transport, under blue ice (<4°C), back to the laboratory. At least one field blank is returned to the laboratory with each group of

samples. A field blank is treated exactly like a sample except that no air is drawn through the cartridge. Samples are stored at $<4^{\circ}\text{C}$ or below until analyzed in the laboratory. Extraction must occur within 7 days of sampling and analysis within 40 days of extraction. Refer to ASTM D4861-94 (1), Appendix X3 for storage stability for various common pesticides and other compounds on PUF or PUF/Tenax TA sandwich.

11. Sample Extraction Procedure

[Note: Sample extraction should be performed under a properly ventilated hood.]

11.1 Sample Extraction

11.1.1 All samples should be extracted within 1 week after collection. All samples should be stored at $<4^{\circ}\text{C}$ until extracted.

11.1.2 All glassware should be washed with a suitable detergent; rinsed with deionized water, acetone, and hexane; rinsed again with deionized water; and fired in an oven (500°C).

11.1.3 Prepare a spiking solution for determination of extraction efficiency. The spiking solution should contain one or more surrogate compounds that have chemical structures and properties similar to those of the analytes of interest. Octachloronaphthalene (OCN) and dibutylchlorodate have been used as surrogates for determination of organochlorine pesticides by GC with an ECD. Tetrachloro-m-xylene and decachlorobiphenyl can also be used together to insure recovery of early and late eluting compounds. For organophosphate pesticides, tributylphosphate or triphenylphosphate may be employed as surrogates. The surrogate solution should be prepared so that addition of 100 μL into the PUF plug results in an extract containing the surrogate compound at the high end of the instrument's calibration range. As an example, the spiking solution for OCN is prepared by dissolving 10 mg of OCN in 10 mL of 10% acetone in n-hexane, followed by serial dilution n-hexane to achieve a final spiking solution of OCN of 1 $\mu\text{g/mL}$.

[Note: Use the recoveries of the surrogate compounds to monitor for unusual matrix effects and gross sample processing errors. Evaluate surrogate recovery for acceptance by determining whether the measured concentration falls within the acceptance limits of 60-120 percent.]

11.1.4 The extracting solution (5% diethyl ether/hexane) is prepared by mixing 900 mL of freshly opened hexane and 100 mL of freshly opened diethyl ether (preserved with ethanol) to a flask.

11.1.5 All clean glassware, forceps, and other equipment to be used should be rinsed with 5% diethyl ether/hexane and placed on rinsed (5% diethyl ether/hexane) aluminum foil until use. The condensing towers should also be rinsed with 5% diethyl ether/hexane. Then add 300 mL of 5% diethyl ether/hexane to the 500 mL round bottom boiling flask and add up to three boiling granules.

11.1.6 Using precleaned (i.e., 5% diethyl ether/hexane Soxhlet extracted) cotton gloves, the glass PUF cartridges are removed from the sealed container, the PUF removed from the glass container and is placed into the 300 mL Soxhlet extractor using prerinsed forceps.

[Note: If "sandwich" trap is used, carefully clean outside walls of cartridge with hexane-soaked cotton swabs or laboratory tissues (discard) and place cartridge into extractor with intake (large end) downward.]

11.1.7 Before extraction begins, add 100 μL of the OCN solution directly to the top of the PUF plug.

[Note: Incorporating a known concentration of the solution onto the sample provides a quality assurance check to determine recovery efficiency of the extraction and analytical processes.]

11.1.8 Connect the Soxhlet extractor to the 500 mL boiling flask and condenser. Wet the glass joints with 5% diethyl ether/hexane to ensure a tight seal between the fittings. If necessary, the PUF plug can be adjusted using forceps to wedge it midway along the length of the siphon. The above procedure should be followed for all samples, with the inclusion of a blank control sample.

11.1.9 The water flow to the condenser towers of the Soxhlet extraction assembly should be checked and the heating unit turned on. As the samples boil, the Soxhlet extractors should be inspected to ensure that they are filling and siphoning properly (4 to 6 cycles/hour). Samples should cycle for a minimum of 16 hours.

11.1.10 At the end of the extracting process (minimum of 16 hours), the heating unit is turned off and the sample cooled to room temperature.

11.1.11 The extracts are then concentrated to 5 mL using a Kuderna-Danish (K-D) apparatus. The K-D is set up, assembled with concentrator tubes, and rinsed. The lower end of the filter tube is packed with glass wool and filled with sodium sulfate to a depth of 40 mm. The filter tube is then placed in the neck of the K-D. The Soxhlet extractors and boiling flasks are carefully removed from the condenser towers and the remaining solvent is drained into each boiling flask. Sample extract is carefully poured through the filter tube into the K-D. Each boiling flask is rinsed three times by swirling hexane along the sides. Once the sample has drained, the filter tube is rinsed down with hexane. Each Synder column is attached to the K-D and rinsed to wet the joint for a tight seal. The complete K-D apparatus is placed on a steam bath and the sample is evaporated to approximately 5 mL.

[Note: Do not allow samples to evaporate to dryness.]

Remove sample from the steam bath, rinse Synder column with minimum of hexane, and allow to cool. Adjust sample volume to 10 mL in a concentrator tube, close with glass stopper and seal with TFE fluorocarbon tape. Alternatively, the sample may be quantitatively transferred (with concentrator tube rinsing) to prescored vials and brought up to final volume. Concentrated extracts are stored at <4°C until analyzed. Analysis should occur no later than 40 days after sample extraction.

11.2 Sample Cleanup

11.2.1 If polar compounds (from example, organophosphorus and carbamate classes) that interfere with GC/ECD analysis are present, use column chromatographic cleanup or alumina. The sample cleanup will permit the analysis of most organochlorine pesticides or PCBs.

11.2.2 Before cleanup, the sample extract is carefully reduced to 1 mL using a gentle stream of clean nitrogen.

11.2.3 A glass chromatographic column (2-mm I.D. x 15-cm long) is packed with alumina, activity grade IV, and rinsed with approximately 20 mL of n-hexane. The concentrated sample extract is placed on the column and eluted with 10 mL of n-hexane at a rate of 0.5 mL/minute. The eluate volume is adjusted to exactly 10 mL and analyzed as per Section 12.

11.2.4 If both PCBs and organochlorine pesticides are sought, alternate cleanup procedures (5,6) may be required (i.e., silicic acid).

11.2.5 Finally, class separation and improved specificity can be achieved by column clean-up and separation on Florisil (6).

12. Analytical Procedure

12.1 Analysis of Organochlorine Pesticides by Capillary Gas Chromatography with Electron Capture Detector (GC/ECD)

[Note: Organochlorine pesticides, PCBs and many nonchlorinated pesticides are responsive to electron capture detection (see Table 1). Most of these compounds can be analyzed at concentration of 1 to 50 ng/mL by GC/ECD. The following procedure is appropriate. Analytical methods that have been used to determine pesticides and PCBs collected from air by this procedure have been published (12).]

12.1.1 Select GC column (e.g., 0.3-mm by 30-m DB-5 column) and appropriate GC conditions to separate the target analytes. Typical operating parameters for this column with splitless injection are: Carrier gas-chromatography grade helium at a flow rate of 1 to 2 mL/min and a column head pressure of 7 to 9 psi (48 to 60 kPa); injector temperature of 250°C; detector temperature of 350°C; initial oven temperature of 50°C held for 2.0 min., ramped at 15°C/min to 150°C for 8 min, ramped at 10°C/min to 295°C then held for 5 min; purge time of 1.0 min. A typical injection volume is 2 to 3 μ L.

12.1.2 Remove sample extract from the refrigerator and allow to warm to room temperature.

12.1.3 Prepare standard solution from reference materials of known purity. Analytically pure standards of organochlorine pesticides and PCBs are available from several commercial sources.

12.1.4 Use the standard solutions of the various compounds of interest to determine relative retention times (RRTs) to an internal standard such as p,p'-DDE, aldrin or octachloronaphthalene. Use 1 to 3- μ L injections or other appropriate volumes.

12.1.5 Determine detector linearity by injecting standard solutions of three different concentrations (amounts) that bracket the range of analyses. The calibration is considered linear if the relative standard deviation (RSD) of the response factors for the three standards is 20 percent or less.

12.1.6 Calibrate the system with a minimum of three levels of calibration standards in the linear range. The low standard should be near the analytical method detection limit. The calibration is considered linear if the relative standard deviation (RSD) of the response factors for the three standards is 20 percent or less. The initial calibration should be verified by the analysis of a standard from an independent source. Recovery of 85 to 115 percent is acceptable. The initial calibration curve should be verified at the beginning of each day and after every ten samples by the analysis of the mid point standard; an RPD of 15% or less is acceptable for continuing use of the initial calibration curve.

12.1.7 Inject 1 to 3 μ L of the sample extract. Record volume injected to the nearest 0.05 μ L.

12.1.8 A typical ECD response for a mixture of single component pesticides using a capillary column is illustrated in Figure 6. If the response (peak height or area) exceeds the calibration range, dilute the extract and reanalyze.

12.1.9 Quantify PCB mixtures by comparison of the total heights or areas of GC peaks (minimum of 5) with the corresponding peaks in the best-matching standard. Use Aroclor 1242 for early-eluting PCBs and either Aroclor 1254 or Aroclor 1260 as appropriate for late-eluting PCBs.

12.1.10 If both PCBs and organochlorine pesticides are present in the same sample, use column chromatographic separation on silicic acid (5,6) prior to GC analysis.

12.1.11 If polar compounds are present that interfere with GC/ECD analysis, use column chromatographic cleanup or alumina, activity grade IV, in accordance with Section 11.2.

12.1.12 For confirmation use a second GC column such as DB-608. All GC procedures except GC/MS require second column confirmation.

12.1.13 For improved resolution use a capillary column such as an 0.25-mm I.D. x 30-m DB-5 with 0.25 μ m film thickness. The following conditions are appropriate.

- Helium carrier gas at 1 mL/min.
- Column temperature program, 90°C (4 min)/16°C/min to 154°C/4°C/min to 270°C.
- Detector, ^{63}Ni ECD at 350°C.
- Make up gas, nitrogen, or 5% methane/95% argon at 60 mL/min.
- Splitless injection, 2 μ L maximum.
- Injector temperature, 220°C.

12.1.14 Class separation and improved specificity can be achieved by column chromatographic separation on Florisil (6).

12.2 Analysis of Organophosphorus Pesticides by Capillary Gas Chromatography with Flame Photometric or Nitrogen-Phosphorus Detectors (GC/FPD/NPD)

[Note: Organophosphorus pesticides are responsive to flame photometric and nitrogen-phosphorus (alkali flame ionization) detection. Most of these compounds can be analyzed at concentrations of 50 to 500 ng/mL using either of these detectors.]

12.2.1 Procedures given in Section 12.1.1 through 12.1.9 and Section 12.1.13 through 12.1.14 apply, except for the selection of surrogates.

12.2.2 Use tributylphosphate, triphenylphosphate, or other suitable compound(s) as surrogates to verify extraction efficiency and to determine RRTs.

12.3 Analysis of Carbamate and Urea Pesticides by Capillary Gas Chromatography with Nitrogen-Phosphorus Detector

12.3.1 Trazine, carbamate, and urea pesticides may be determined by capillary GC (DB-5, DB-17, or DB-1701 stationary phase) using nitrogen-phosphorus detection or MS-SIM with detection limits in the 0.05 to 0.2 μ L/mL range. Procedures given in Section 12.1.1 through 12.1.9 and Section 12.1.13 through 12.1.14 apply, except for the selection of surrogates, detector, and make up gas.

12.3.2 Thermal degradation may be minimized by reducing the injector temperature to 200°C. HPLC may also be used, but detection limits will be higher (1 to 5 μ g/mL).

12.3.3 N-methyl carbamates may be determined using reverse-phase high performance liquid chromatography (HPLC) (C-18) (Section 12.4) and post-column derivatization with o-phthaldehyde and fluorescence detection (EPA Method 531). Detection limits of 0.01 to 0.1 μ g/mL can be achieved.

12.4 Analysis of Carbamate, Urea, Pyrethroid, and Phenolic Pesticides by High Performance Liquid Chromatography (HPLC)

[Note: Many carbamate pesticides, urea pesticides, pyrethrins, phenols, and other polar pesticides may be analyzed by high HPLC with fixed or variable wavelength UV detection. Either reversed-phase or normal phase chromatography may be used. Detection limits are 0.2 to 10 μ g/mL of extract.]

12.4.1 Select HPLC column (i.e., Zorbax-SIL, 46-mm I.D. x 25-cm, or μ -Bondapak C18, 3.9-mm x 30-cm, or equivalent).

12.4.2 Select solvent system (i.e., mixtures of methanol or acetonitrile with water or mixtures of heptane or hexane with isopropanol).

12.4.3 Follow analytical procedures given in Sections 12.1.2 through 12.1.9.

12.4.4 If interferences are present, adjust the HPLC solvent system composition or use column chromatographic clean-up with silica gel, alumina, or Florisil (6).

12.4.5 An electrochemical detector may be used to improve sensitivity for some ureas, carbamates, and phenolics. Much more care is required in using this detector, particularly in removing dissolved oxygen from the mobile phase and sample extracts.

12.4.6 Chlorophenol (di- through penta-) may be analyzed by GC/ECD or GC/MS after derivatization with pentafluorobenzylbromide (EPA Method 604).

12.4.7 Chlorinated phenoxyacetic acid herbicides and pentachlorophenol can be analyzed by GC/ECD or GC/MS after derivatization with diazomethane (EPA Method 515). DB-5 and DB-1701 columns (0.25-mm I.D. x 30-m) at 60 to 300°C/4°C per min have been found to perform well.

12.5 Analysis of Pesticides and PCBs by Gas Chromatography with Mass Spectrometry Detection (GC/MS)

[Note: A mass spectrometer operating in the selected ion monitoring mode is useful for confirmation and identification of pesticides.]

12.5.1 A mass spectrometer operating in the select ion monitoring (SIM) mode can be used as a sensitive detector for multi-residue determination of a wide variety of pesticides. Mass spectrometers are now available that provide detection limits comparable to nitrogen-phosphorus and electron capture detectors.

12.5.2 Most of the pesticides shown in Table 1 have been successfully determined by GC/MS/SIM. Typical GC operating parameters are as described in Section 12.1.1.

12.5.3 The mass spectrometer is typically operated using positive ion electron impact ionization (70 eV). Other instrumental parameters are instrument specific.

12.5.4 p-Terphenyl-d₁₄ is commonly used as a surrogate for GC/MS analysis.

12.5.5 Quantification is typically performed using an internal standard method. 1,4-Dichlorobenzene, naphthalene-d₈, acenaphthene-d₁₀, phenanthrene-d₁₀, chrysene-d₁₂ and perylene-d₁₂ are commonly used as internal standards. Procedures given in Section 12.1.1 through 12.1.9 and Section 12.1.13 through 12.1.14 apply, except for the selection of surrogates, detector, and make up gas.

12.5.6 See ASTM Practice D 3687 for injection technique, determination of relative retention times, and other procedures pertinent to GC and HPLC analyses.

12.6 Sample Concentration

12.6.1 If concentrations are too low to detect by the analytical procedure of choice, the extract may be concentrated to 1 mL or 0.5 mL by carefully controlled evaporation under an inert atmosphere. The following procedure is appropriate.

12.6.2 Place K-D concentrator tube in a water bath and analytical evaporator (nitrogen blow-down) apparatus. The water bath temperature should be from 25°C to 50°C.

12.6.3 Adjust nitrogen flow through hypodermic needle to provide a gentle stream.

12.6.4 Carefully lower hypodermic needle into the concentrator tube to a distance of about 1 cm above the liquid level.

12.6.5 Continue to adjust needle placement as liquid level decreases.

12.6.6 Reduce volume to slightly below desired level.

12.6.7 Adjust to final volume by carefully rinsing needle tip and concentrator tube well with solvent (usually n-hexane).

13. Calculations

13.1 Determination of Concentration

13.1.1 The concentration of the analyte in the extract solution can be taken from a standard curve where peak height or area is plotted linearly against concentration in nanograms per milliliter (ng/mL). If the detector response is known to be linear, a single point is used as a calculation constant.

13.1.2 From the standard curve, determine the nanograms of analyte standard equivalent to the peak height or area for a particular compound.

13.1.3 Ascertain whether the field blank is contaminated. Blank levels should not exceed 10 ng/sample for organochlorine pesticides or 100 ng/sample for PCBs and other pesticides. If the blank has been contaminated, the sampling series must be held suspect.

13.1.4 Quantity of the compound in the sample (A) is calculated using the following equation:

$$A = 1000 \left(\frac{A_s \times V_e}{V_i} \right)$$

where:

A = total amount of analyte in the sample, ng.

A_s = calculated amount of material injected onto the chromatograph based on calibration curve for injected standards, ng.

V_e = final volume of extract, mL.

V_i = volume of extract injected, μ L.

1000 = factor for converting microliters to milliliters.

13.1.5 The extraction efficiency (EE) is determined from the recovery of surrogate spike as follows:

$$EE(\%) = \left| \frac{S}{S_a} \right| [100]$$

where:

EE = extraction efficiency, %.

S = amount of spike recovered, ng.

S_a = amount of spike added to plug, ng.

The extraction efficiency (surrogate recovery) must fall between 60-120% to be acceptable.

13.1.6 The total volume of air sampled under ambient conditions is determined using the following equation:

$$V_a = \frac{\sum_{i=1}^n (T_i \times F_i)}{1000 \text{ L/m}^3}$$

where:

V_a = total volume of air sampled, m^3 .

T_i = length of sampling segment between flow checks, min.

F_i = average flow during sampling segment, L/min.

13.1.7 The air volume is corrected to EPA standard temperature (25°C) and standard pressure (760 mm Hg) as follows:

$$V_s = V_a \left(\frac{P_b - P_w}{760 \text{ mm Hg}} \right) \left(\frac{298\text{K}}{t_A} \right)$$

where:

V_s = volume of air at standard conditions (25°C and 760 mm Hg), std. m^3 .

V_a = total volume of air sampled, m^3 .

P_b = average ambient barometric pressure, mm Hg.

P_w = vapor pressure of water at calibration temperature, mm Hg.

t_A = average ambient temperature, $^\circ\text{C} + 273$.

13.1.8 If the proper criteria for a sample have been met, concentration of the compound in a standard cubic meter of air sampled is calculated as follows:

$$C_a(\text{ng/std. m}^3) = \left[\frac{(A)}{(V_s)} \right] \left[\frac{(100)}{(SE(\%))} \right]$$

where:

SE = sampling efficiency as determined by the procedure outlined in Section 14.

If it is desired to convert the air concentration value to parts per trillion (ppt) in dry air at standard temperature and pressure (STP), the following conversion is used:

$$\text{ppt} = 0.844 (C_a)$$

The air concentration can be converted to parts per trillion (v/v) in air at STP as follows:

$$\text{pptv} = \left[\frac{(24.45) (C_a)}{(MW)} \right]$$

where:

MW = molecular weight of the compound of interest, g/g-mole.

13.1.9 If quantification is performed using an internal standard, a relative response factor (RRF) is calculated by the equation:

$$RRF = \frac{(I_s)(C_{is})}{(I_{is})(C_s)}$$

where:

I_s = integrated area of the target analyte peak, counts.

I_{is} = integrated area of the internal standard peak, counts.

C_{is} = concentration of the internal standard, ng/ μ L.

C_s = concentration of the analyte, ng/ μ L.

13.1.10 The concentration of the analyte (C_a) in the sample is then calculated as follows:

$$C_a = \frac{(I_s)(C_{is})}{(RRF)(I_{is})}$$

where:

C_a = concentration of analyte, ng/ m^3

I_s = integrated area of the target analyte peak, counts.

RRF = relative response factor (see Section 13.1.10).

14. Sampling and Retention Efficiencies

14.1 General

14.1.1 Before using Compendium Method TO-10A, the user should determine the sampling efficiency for the compound of interest. The sampling efficiencies shown in Tables 2, 3, 4, and 5 were determined for approximately 1 m^3 of air at about 25°C, sampled at 3.8 L/min. The SE values in these tables may be used for similar sampling conditions; for other compounds or conditions, SE values must be determined.

14.1.2 Sampling efficiencies for the pesticides shown in Table 6 are for a flowrate of 3.8 L/min and at 25°C. For compounds not listed, longer sampling times, different flow rates, or other air temperatures, the following procedure may be used to determine sampling efficiencies.

14.2 Determining SE

14.2.1 SE is determined by a modified impinger assembly attached to the sampler pump, as illustrated in Figure 7. A clean PUF is placed in the pre-filter location and the inlet is attached to a nitrogen line.

[Note: Nitrogen should be used instead of air to prevent oxidation of the compounds under test. The oxidation would not necessarily reflect what may be encountered during actual sampling and may give misleading sampling efficiencies.]

Two PUF plugs (22-mm x 7.6-cm) are placed in the primary and secondary traps and are attached to the pump.

14.2.2 A standard solution of the compound of interest is prepared in a volatile solvent (i.e., hexane, pentane, or benzene). A small, accurately measured volume (i.e., 1 mL) of the standard solution is placed into the modified midjet impinger. The sampler pump is set at the rate to be used in field application and then activated. Nitrogen is drawn through the assembly for a period of time equal to or exceeding that intended for field application. After the desired sampling test period, the PUF plugs are removed and analyzed separately as per Section 12.

14.2.3 The impinger is rinsed with hexane or another suitable solvent and quantitatively transferred to a volumetric flask or concentrator tube for analysis.

14.2.4 The sampling efficiency (SE) is determined using the following equation:

$$\% \text{ SE} = \frac{W_1}{W_0 - W_r} \times 100$$

where:

W_1 = amount of compound extracted from the primary trap, ng.

W_0 = original amount of compound added to the impinger, ng.

W_r = residue left in the impinger at the end of the test, ng.

14.2.5 If material is found in the secondary trap, it is an indication that breakthrough has occurred. The addition of the amount found in the secondary trap, W_2 , to W_1 , will provide an indication for the overall sampling efficiency of a tandem-trap sampling system. The sum of W_1 , W_2 (if any), and W_r must equal (approximately $\pm 10\%$) W_0 or the test is invalid.

14.2.6 If the compound of interest is not sufficiently volatile to vaporize at room temperature, the impinger may be heated in a water bath or other suitable heater to a maximum of 50°C to aid volatilization. If the compound of interest cannot be vaporized at 50°C without thermal degradation, dynamic retention efficiency (RE_d) may be used to estimate sampling efficiency. Dynamic retention efficiency is determined in the manner described in Section 14.2.7. Table 7 lists those organochlorine pesticides which dynamic retention efficiencies have been determined.

14.2.7 A pair of PUF plugs is spiked by slow, dropwise addition of the standard solution to one end of each plug. No more than 0.5 to 1 mL of solution should be used. Amounts added to each plug should be as nearly the same as possible. The plugs are allowed to dry for 2 hours in a clean, protected place (i.e., desiccator). One spiked plug is placed in the primary trap so that the spiked end is at the intake and one clean unspiked plug is placed in the secondary trap. The other spiked plug is wrapped in hexane-rinsed aluminum foil and stored in a clean place for the duration of the test (this is the static control plug, Section 14.2.8). Prefiltered nitrogen or ambient air is drawn through the assembly as per Section 14.2.2.

[Note: Impinger may be discarded.]

Each PUF plug (spiked and static control) is analyzed separately as per Section 12.

14.2.8 This dynamic retention efficiency ($\% RE_d$) is calculated as follows:

$$\% RE_d = \frac{W_1}{W_0} \times 100$$

where:

W_1 = amount of compound recovered from primary plug, ng.

W_o = amount of compound added to primary plug, ng.

If a residue, W_2 , is found on the secondary plug, breakthrough has occurred. The sum of $W_1 + W_2$ must equal W_o , within 25% or the test is invalid. For most compounds tested by this procedure, % RE_d values are generally less than % SE values determined per Section 14.2. The purpose of the static RE_d determination is to establish any loss or gain of analyte unrelated to the flow of nitrogen or air through the PUF plug.

15. Performance Criteria and Quality Assurance

[Note: This section summarizes required quality assurance (QA) measures and provides guidance concerning performance criteria that should be achieved within each laboratory.]

15.1 Standard Operating Procedures (SOPs)

15.1.1 Users should generate SOPs describing the following activities accomplished in their laboratory: (1) assembly, calibration, and operation of the sampling system, with make and model of equipment used; (2) preparation, purification, storage, and handling of sampling cartridges; (3) assembly, calibration, and operation of the analytical system, with make and model of equipment used; and (4) all aspects of data recording and processing, including lists of computer hardware and software used.

15.1.2 SOPs should provide specific stepwise instructions and should be readily available to, and understood by, the laboratory personnel conducting the work.

15.2 Process, Field, and Solvent Blanks

15.2.1 One PUF cartridge from each batch of approximately twenty should be analyzed, without shipment to the field, for the compounds of interest to serve as a process blank.

15.2.2 During each sampling episode, at least one PUF cartridge should be shipped to the field and returned, without drawing air through the sampler, to serve as a field blank.

15.2.3 Before each sampling episode, one PUF plug from each batch of approximately twenty should be spiked with a known amount of the standard solution. The spiked plug will remain in a sealed container and will not be used during the sampling period. The spiked plug is extracted and analyzed with the other samples. This field spike acts as a quality assurance check to determine matrix spike recoveries and to indicate sample degradation.

15.2.4 During the analysis of each batch of samples, at least one solvent process blank (all steps conducted but no PUF cartridge included) should be carried through the procedure and analyzed.

15.2.5 All blank levels should not exceed 10 ng/sample for single components or 100 ng/sample for multiple component mixtures (i.e., for organochlorine pesticides and PCBs).

15.3 Sampling Efficiency and Spike Recovery

15.3.1 Before using the method for sample analysis, each laboratory must determine its sampling efficiency for the component of interest as per Section 14.

15.3.2 The PUF in the sampler is replaced with a hexane-extracted PUF. The PUF is spiked with a microgram level of compounds of interest by dropwise addition of hexane solutions of the compounds. The solvent is allowed to evaporate.

15.3.3 The sampling system is activated and set at the desired sampling flow rate. The sample flow is monitored for 24 hours.

15.3.4 The PUF cartridge is then removed and analyzed as per Section 12.

15.3.5 A second sampler, unspiked, is collected over the same time period to account for any background levels of components in the ambient air matrix.

15.3.6 In general, analytical recoveries and collection efficiencies of 75% are considered to be acceptable method performance.

15.3.7 Replicate (at least triplicate) determinations of collection efficiency should be made. Relative standard deviations for these replicate determinations of $\pm 15\%$ or less are considered acceptable performance.

15.3.8 Blind spiked samples should be included with sample sets periodically as a check on analytical performance.

15.4 Method Precision and Bias

15.4.1 Precision and bias in this type of analytical procedure are dependent upon the precision and bias of the analytical procedure for each compound of concern, and the precision and bias of the sampling process.

15.4.2 Several different parameters involved in both the sampling and analysis steps of this method collectively determine the precision and bias with which each compound is detected. As the volume of air sampled is increased, the sensitivity of detection increases proportionately within limits set by: (a) the retention efficiency for each specific component trapped on the polyurethane foam plug, and (b) the background interference associated with the analysis of each specific component at a given site sampled. The sensitivity of detection of samples recovered by extraction depends on: (a) the inherent response of the particular GC detector used in the determinative step, and (b) the extent to which the sample is concentrated for analysis. It is the responsibility of the analyst(s) performing the sampling and analysis steps to adjust parameters so that the required detection limits can be obtained.

15.4.3 The reproducibility of this method for most compounds for which it has been evaluated has been determined to range from ± 5 to $\pm 30\%$ (measured as the relative standard deviation) when replicate sampling cartridges are used ($N > 5$). Sample recoveries for individual compounds generally fall within the range of 90 to 110%, but recoveries ranging from 65 to 125% are considered acceptable. PUF alone may give lower recoveries for more volatile compounds (i.e., those with saturation vapor pressures $> 10^{-3}$ mm Hg). In those cases, another sorbent or a combination of PUF and Tenax TA (see Figure 2) should be employed.

15.5 Method Safety

15.5.1 This procedure may involve hazardous materials, operations, and equipment. This method does not purport to address all of the safety problems associated with its use.

15.5.2 It is the user's responsibility to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to the implementation of this procedure. This should be part of the user's SOP manual.

16. References

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TABLE 1. COMPOUNDS FOR WHICH PROCEDURE HAS BEEN TESTED¹

Compound	Recommended Analysis ²	Compound	Recommended Analyses
Alachlor	GC/ECD	Heptachlor	GC/ECD
Aldrin	GC/ECD	Heptachlor epoxide	GC/ECD
Allethrin	HPLC/UV	Hexachlorobenzene	GC/ECD
Aroclor 1242	GC/ECD	Hexachlorocyclopentadiene ^{3,4}	GC/ECD
Aroclor 1254	GC/ECD	Lindane (γ -BHC)	GC/ECD
Aroclor 1260	GC/ECD	Linuron	HPLC/UV
Atrazine	GC/NPD	Malathion	GC/NPD or FPD
Bendiocarb	HPLC/UV	Methyl parathion	GC/NPD or FPD
BHC (α - and β -Hexachlorocyclohexanes)	GC/ECD	Methoxychlor	GC/FCD
Captan	GC/ECD	Metolachlor	GC/ECD
Carbaryl	HPLC/UV	Mexacarbate	GC/FCD
Carbofuran	HPLC/UV	Mirex	GC/ECD
Chlordane, technical	GC/ECD	Monuron	HPLC/UV
Chlorothalonil	GC/ECD	Trans-nonachlor	GC/ECD
Chlorotoluron	HPLC/UV	Oxychlordane	GC/ECD
Chlorpyrifos	GC/ECD	Pentachlorobenzene	GC/ECD
2,4-D esters and salts	GC/ECD	Pentachlorophenol	GC/ECD
Dacthal	GC/ECD	Permethrin (cis and trans)	HPLC/UV
<i>p,p'</i> -DDT	GC/ECD	<i>o</i> -Phenylphenol	HPLC/UV
<i>p,p'</i> -DDE	GC/ECD	Phorate	GC/NPD or FPD
Diazinon	GC/NPD or FPD	Propazine	GC/NPD
Dicloran	GC/ECD	Propoxur (Baygon)	HPLC/UV
Dieldrin	GC/ECD	Pyrethrin	HPLC/UV
Dichlorvos (DDVP)	GC/ECD	Resmethrin	HPLC/UV
Dicofol	GC/ECD	Ronnel	GC/ECD
Dicrotophos	HPLC/UV	Simazine	HPLC/UV
Diuron	HPLC/UV	Terbuthiuron	HPLC/UV
Ethyl parathion	GC/NPD or FPD	1,2,3,4-tetrachlorobenzene ³	GC/ECD
Fenvalerate	HPLC/UV	1,2,3-trichlorobenzene ³	GC/ECD
Fluometuron	HPLC/UV	2,3,5-trichlorophenol	GC/ECD
Folpet	GC/ECD	Trifluralin	GC/ECD

¹The following recommendations are specific for that analyte for maximum sensitivity.

²GC = gas chromatography; ECD = electron capture detector; FPD = flame photometric detector; HPLC = high performance liquid chromatography; NPD = nitrogen-phosphorus detector; UV = ultraviolet absorption detector, (GC/MS (gas chromatography/mass spectrometry) may also be used).

³Using PUF/Tenax-TA "sandwich" trap.

⁴Compound is very unstable in solution.

TABLE 2. SAMPLING EFFICIENCIES FOR SOME ORGANOCHLORINE PESTICIDES

Compound	Quantity Introduced, μg^2	Air Volume, m^3	Sampling efficiency, %		
			mean	RSD	n
α -Hexachlorocyclohexane (α -BHC)	0.005	0.9	115	8	6
γ -Hexachlorocyclohexane (Lindane)	0.05-1.0	0.9	91.5	8	5
Chlordane, technical	0.2	0.9	84.0	11	8
p,p'-DDT	0.6, 1.2	0.9	97.5	21	12
p,p'-DDE	0.2, 0.4	0.9	102	11	12
Mirex	0.6, 1.2	0.9	85.9	22	7
2,4-D Esters:					
Isopropyl	0.5	3.6	92.0	5	12
Butyl	0.5	3.6	82.0	10	11
Isobutyl	0.5	3.6	79.0	20	12
Isocetyl	0.5	3.6	>80 ²	--	--

¹Air volume = 0.9 m^3 .²Not vaporized. Value base on %RE = 81.0 (RSD = 10%, n = 6).

TABLE 3. SAMPLING EFFICIENCIES FOR ORGANOPHOSPHORUS PESTICIDES

Compound	Quantity Introduced, μg^2	Sampling efficiency, %		
		mean	RSD	n
Dichlorvos (DDVP)	0.2	72.0	13	2
Ronnel	0.2	106	8	12
Chlorpyrifos	0.2	108	9	12
Diazinon ¹	1.0	84.0	18	18
Methyl parathion ¹	0.6	80.0	19	18
Ethyl parathion ¹	0.3	75.9	15	18
Malathion ¹	0.3	100 ³	--	--

¹Analyzed by gas chromatography with nitrogen phosphorus detector or flame photometric detector.²Air volume = 0.9 m^3 .³Decomposed in generator; value based on %RE = 101 (RDS = 7, n = 4).

TABLE 4. SAMPLING EFFICIENCIES FOR SOME SEMI-VOLATILE
ORGANOCHLORINE COMPOUNDS AND PCBs

Compound	Quantity Introduced, μg^1	Sampling efficiency, %		
		mean	RSD	n
1,2,3-Trichlorobenzene	1.0	6.6 ²	22	8
1,2,3,4-Tetrachlorobenzene	1.0	62.3 ²	33	5
Pentachlorobenzene	1.0	94.0	12	5
Hexachlorobenzene	0.5, 1.0	94.5	8	5
Hexachlorocyclopentadiene	1.0	8.3 ²	12	5
2,4,5-Trichlorophenol	1.0	108	3	5
Pentachlorophenol	1.0	107	16	5
Aroclor 1242	0.1	96.0	15	6
Aroclor 1254	0.1	95.0	7	6
Aroclor 1260	0.1	109	5	11

¹ Air volume = 0.9 m³.² % SEs were 98, and 97% (n = 2), respectively, for these three compounds by the PUF/Tenax® TA "sandwich" trap.

TABLE 5. SAMPLING EFFICIENCIES FOR CARBAMATES, UREAS, TRIAZINES, AND PYRETHRINS

Compound	Spike Level, µg/plug	Static Recovery, %		n	Retention Efficiency, %		Sampling Efficiency, %		n
		mean	RSDP		mean	RSD	mean	RSD	
Carbamates:									
Propoxur	5	61.4	10	6	77.6	37	96.7	11	6
Carbofuran	15	55.3	12	6	64.2	46	87.2	14	6
Bendi carb	50	57.3	11	6	69.8	43	62.1	14	6
Mexacarbate	10	62.8	19	6	62.7	41	89.8	14	6
Carbaryl	100	56.6	14	6	63.6	53	0	13	6
Ureas:									
Monuron	19	87.0	6	6	91.2	6	0		
Diuron	20	84.1	8	6	90.0	2	0		
Linuron	20	86.7	8	6	92.5	4	0		
Terbutiuron	18	85.0	8	6	88.8	8	0		
Fluometuron	20	91.4	10	6	101	3	0		
Chlortoluron	20	86.2	11	6	92.0	7	0		
Triazines:									
Simazine	10	103	6	5	101	9	0		
Atrazine	10	104	7	5	98.9	7	0		
Propazine	10	105	11	5	99.9	14	0		
Pyrethrins:									
Pyrethrin I	(9.7)	90.5	10	6	95.6	22	0		
Pyrethrin II	(6.1)	88.6	11	6	69.9	29	0		
Allethrin	25	69.2	9	5	58.3	12	0		
d-trans-Allethrin	25	76.8	9	6	74.4	9	0		
Dicrotophos	25	72.0	22	6	71.7	8	0		
Resmethrin	25	76.5	14	6	66.7	14	0		
Fenvalerate	25	87.9	3	6	57.2	20	0		

TABLE 6. EXTRACTION AND 24-H SAMPLING EFFICIENCIES FOR VARIOUS PESTICIDES AND RELATED COMPOUNDS

Compound	Extraction Efficiency ¹ , %		Sampling Efficiency ² , % at					
			10 ng/m ³		100 ng/m ³		1,000 ng/m ³	
	mean	RSD	mean	RSD	mean	RSD	mean	RSD
Chlorpyrifos	83.3	11.5	83.7	18.0	92.7	15.1	83.7	18.0
Pentachlorophenol	84.0	22.6	66.7	42.2	52.3	36.2	66.7	42.2
Chlordane	95.0	7.1	96.0	1.4	74.0	8.5	96.0	1.4
o-Phenylphenol	47.0	46.7	46.0	19.1	45.3	29.9	46.0	19.1
Lindane	96.0	6.9	91.7	11.6	93.0	2.6	91.7	11.6
DDVP	88.3	20.2	51.0	53.7	106.0	1.4	51.0	53.7
2,4-D Methyl Ester	--	--	75.3	6.8	58.0	23.6	75.3	6.8
Heptachlor	99.0	1.7	97.3	13.6	103.0	17.3	97.3	13.6
Aldrin	97.7	4.0	90.7	5.5	94.0	2.6	90.7	5.5
Dieldrin	95.0	7.0	82.7	7.6	85.0	11.5	82.7	7.6
Ronnel	80.3	19.5	74.7	12.1	60.7	15.5	74.7	12.2
Diazinon	72.0	21.8	63.7	18.9	41.3	26.6	63.7	19.9
trans-Nonachlor	97.7	4.0	96.7	4.2	101.7	15.3	96.7	4.2
Oxychlorodane	100.0	0.0	95.3	9.5	94.3	1.2	95.3	9.5
α-BHC	98.0	3.5	86.7	13.7	97.0	18.2	86.7	13.7
Bendiocarb	81.3	8.4	59.7	16.9	30.7	23.5	59.7	16.9
Chlorothalonil	90.3	8.4	76.7	6.1	70.3	6.5	76.7	6.1
Heptachlor Epoxide	100.0	0.0	95.3	5.5	97.7	14.2	95.3	5.5
Dacthal	--	--	87.0	9.5	95.3	22.2	87.0	9.5
Aroclor 1242	91.7	14.4	95.0	15.5	94.7	17.5	95.0	15.5

¹Mean values for one spike at 550 ng/plug and two spikes at 5,500 ng/plug.²Mean values for three determinations.

TABLE 7. EXTRACTION AND 24-H DYNAMIC RETENTION EFFICIENCIES FOR VARIOUS PESTICIDES AND RELATED COMPOUNDS

Compound	Extraction Efficiency ¹ , %		Sampling Efficiency ² , % at					
			10 ng/m ³		100 ng/m ³		1,000 ng/m ³	
	mean	RSD	mean	RSD	mean	RSD	mean	RSD
Propoxur	77.5	71.4	92.0	--	91.7	22.8	101.0	18.4
Resmethrin	95.5	71.4	79.0	--	100.7	13.1	107.0	4.4
Dicofol	57.0	8.5	38.0	25.9	65.0	8.7	69.0	--
Captan	73.0	12.7	56.0	--	45.5	64.3	84.3	16.3
Carbaryl	74.0	82.0	102.0	--	61.0	--	113.0	6.1
Malathion	76.5	44.5	108.0	--	54.0	16.0	77.3	7.6
cis-Permethrin	88.7	10.3	101.0	28.5	85.0	26.9	89.0	11.3
trans-Permethrin	88.7	11.0	67.3	34.8	80.7	56.4	108.3	9.5
Methoxychlor	65.5	4.9	--	--	--	--	78.5	2.1
Atrazine	75.0	50.5	--	--	73.0	30.1	83.0	9.5
Folpet	86.7	11.7	--	--	78.0	--	93.0	--
Aroclor 1260	92.0	14.5	88.0	9.6	85.3	9.9	107.1	13.6

¹Mean values for one spike at 550 ng/plug and two spikes at 5,500 ng/plug.²Mean values for three determinations.

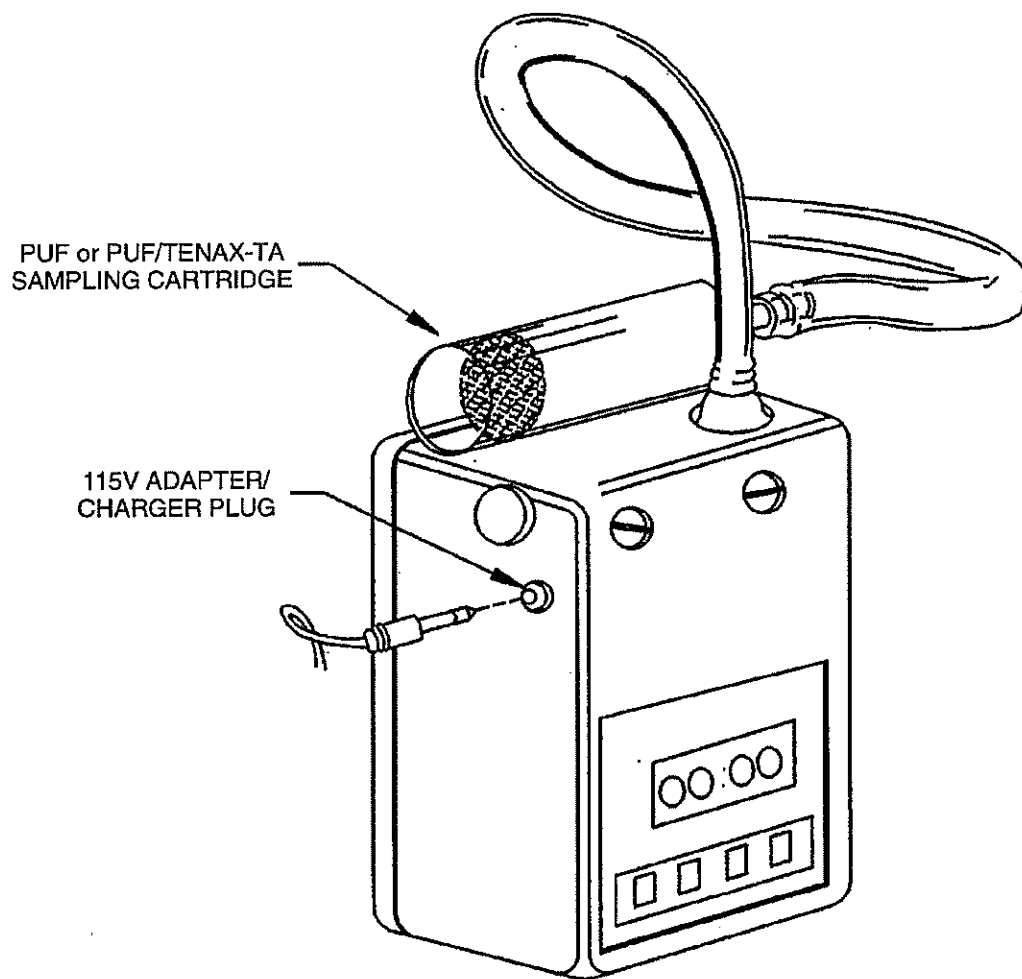


Figure 1. Low volume air sampler.

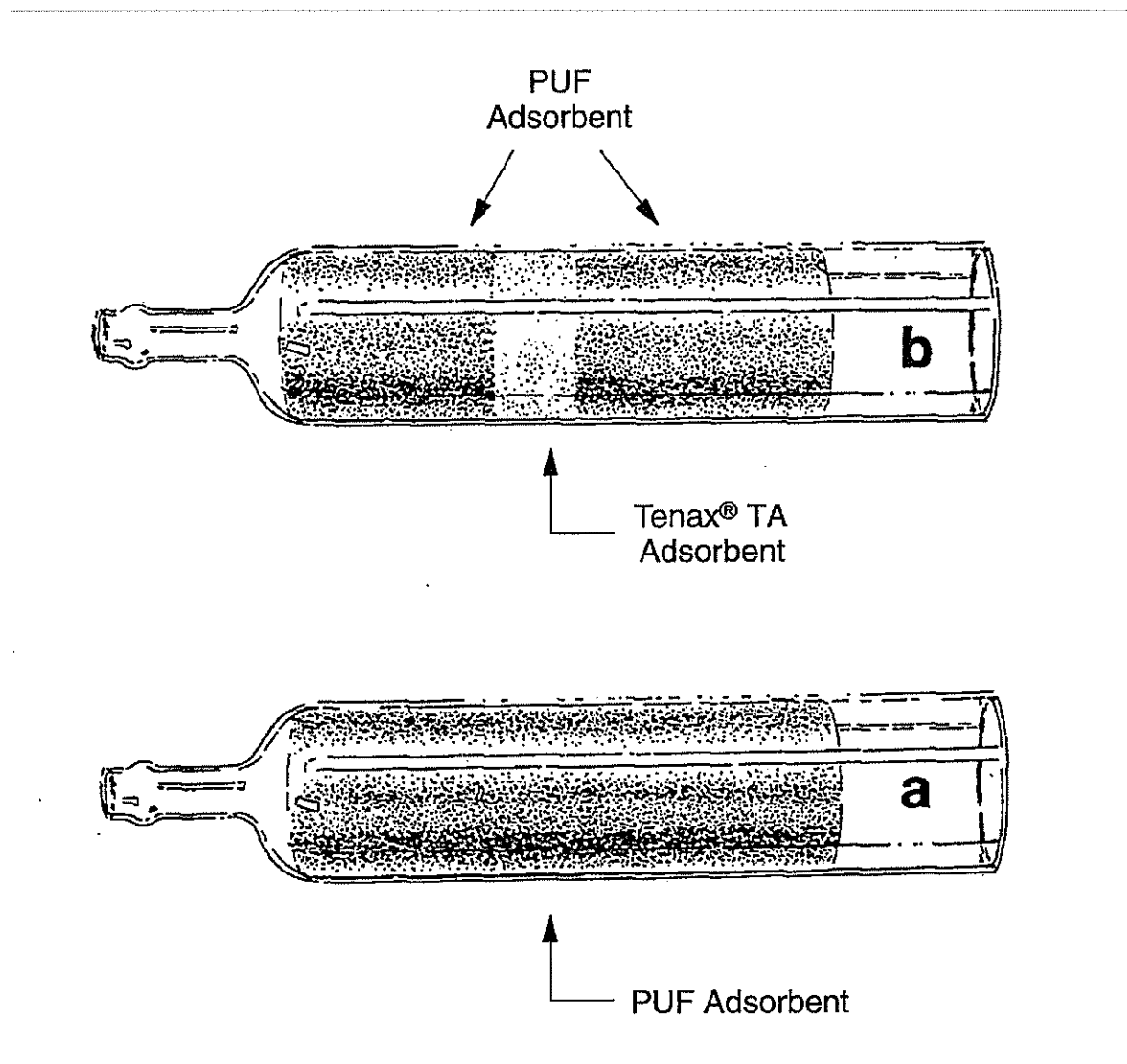


Figure 2. Polyurethane foam (PUF) sampling cartridge (a) and PUF-Tenax® TA "sandwich" sampling cartridge (b).

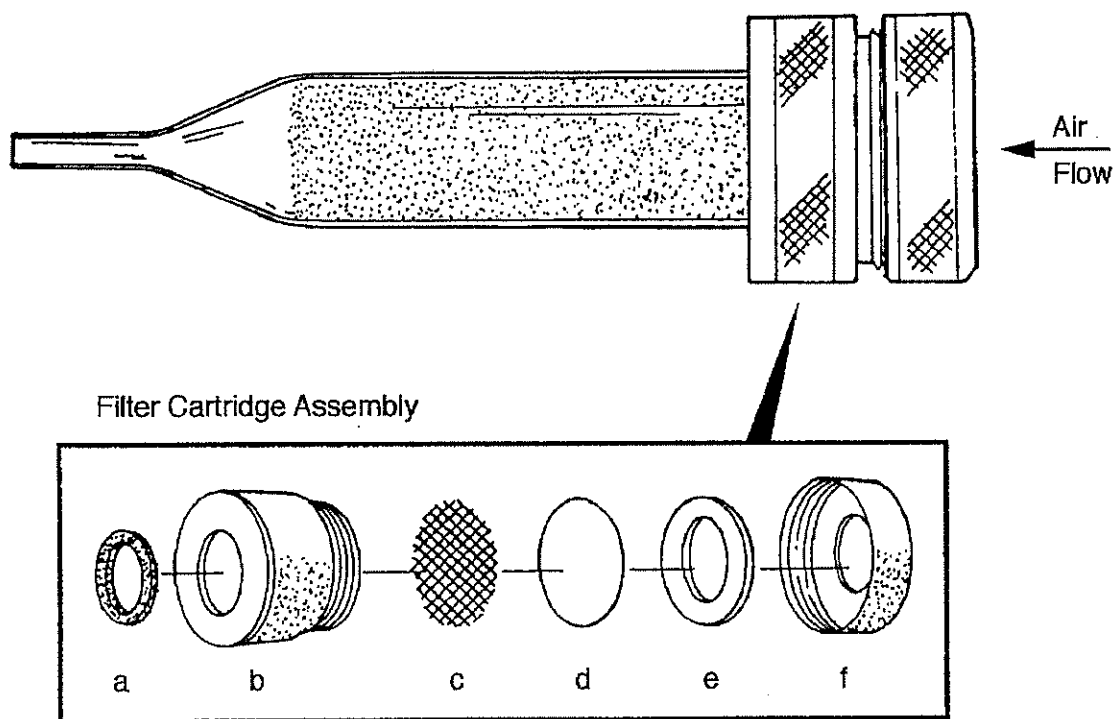


Figure 3. Open-face filter assembly attached to a PUF cartridge:
(a) Inner Viton® o-ring, (b) filter cartridge, (c) stainless steel screen, (d) quartz filter,
(e) filter ring, and (f) cartridge screw cap.

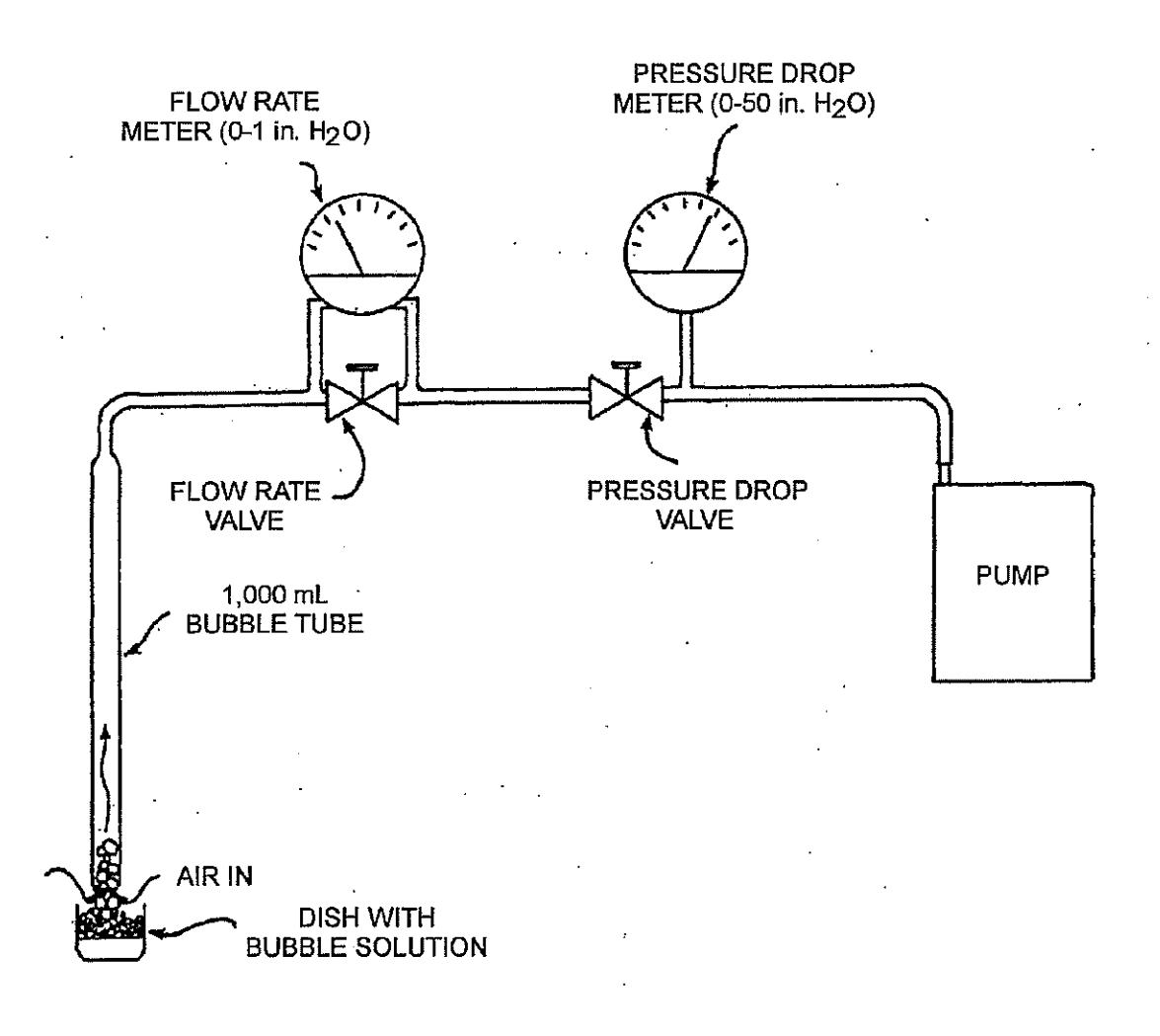


Figure 4. Calibration assembly for air sampler pump.

COMPENDIUM METHOD TO-10A FIELD TEST DATA SHEET (FTDS)

I. GENERAL INFORMATION

PROJECT: _____ DATE(S) SAMPLED: _____
 SITE: _____ TIME PERIOD SAMPLED: _____
 LOCATION: _____ OPERATOR: _____
 INSTRUMENT MODEL NO.: _____ CALIBRATED BY: _____
 PUMP SERIAL NO.: _____ RAIN: ____YES ____NO

ADSORBENT CARTRIDGE INFORMATION:

	Cartridge 1	Cartridge 2	Cartridge 3	Cartridge 4
Type:	_____	_____	_____	_____
Adsorbent:	_____	_____	_____	_____
Serial No.:	_____	_____	_____	_____
Sample No.:	_____	_____	_____	_____

II. SAMPLING DATA

Cartridge Identifi- cation	Sampling Location	Ambient Temp., °F	Ambient Pressure, in Hg	Flow Rate (Q), mL/min		Sampling Period		Total Sampling Time, min.	Total Sample Volume, L
				Cartridge 1	Cartridge 2	Start	Stop		

III. FIELD AUDIT

	<u>Cartridge 1</u>	<u>Cartridge 2</u>	<u>Cartridge 3</u>	<u>Cartridge 4</u>
Audit Flow Check Within _____	_____	_____	_____	_____
10% of Set Point (Y/N)?	pre- _____	pre- _____	pre- _____	pre- _____
	post- _____	post- _____	post- _____	post- _____

CHECKED BY: _____

DATE: _____

Figure 5. Compendium Method TO-10A field test data sheet.

OPERATING CONDITIONS

Column Type: DB-5 0.32 capillary,
0.25 μ m film thickness

Column Temperature Program: 90°C(4min)/16°C per min to
154°C/4°C per min to 270°C.

Detector: Electron Capture

Carrier Gas: Helium at 1 mL/min.

Make Up Gas: 5% Methane/95% Argon at 60 mL/min.

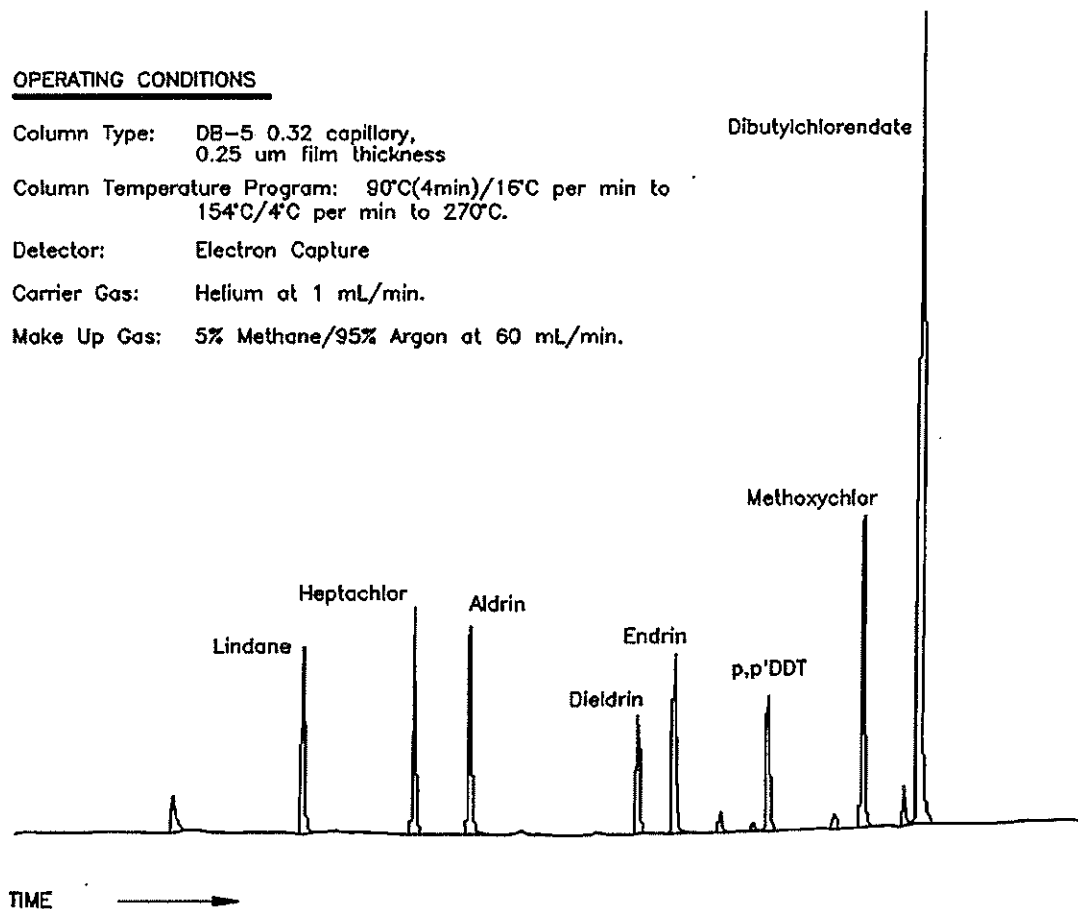


Figure 6. Chromatogram showing a mixture of single component pesticides determined by GC/ECD using a capillary column.

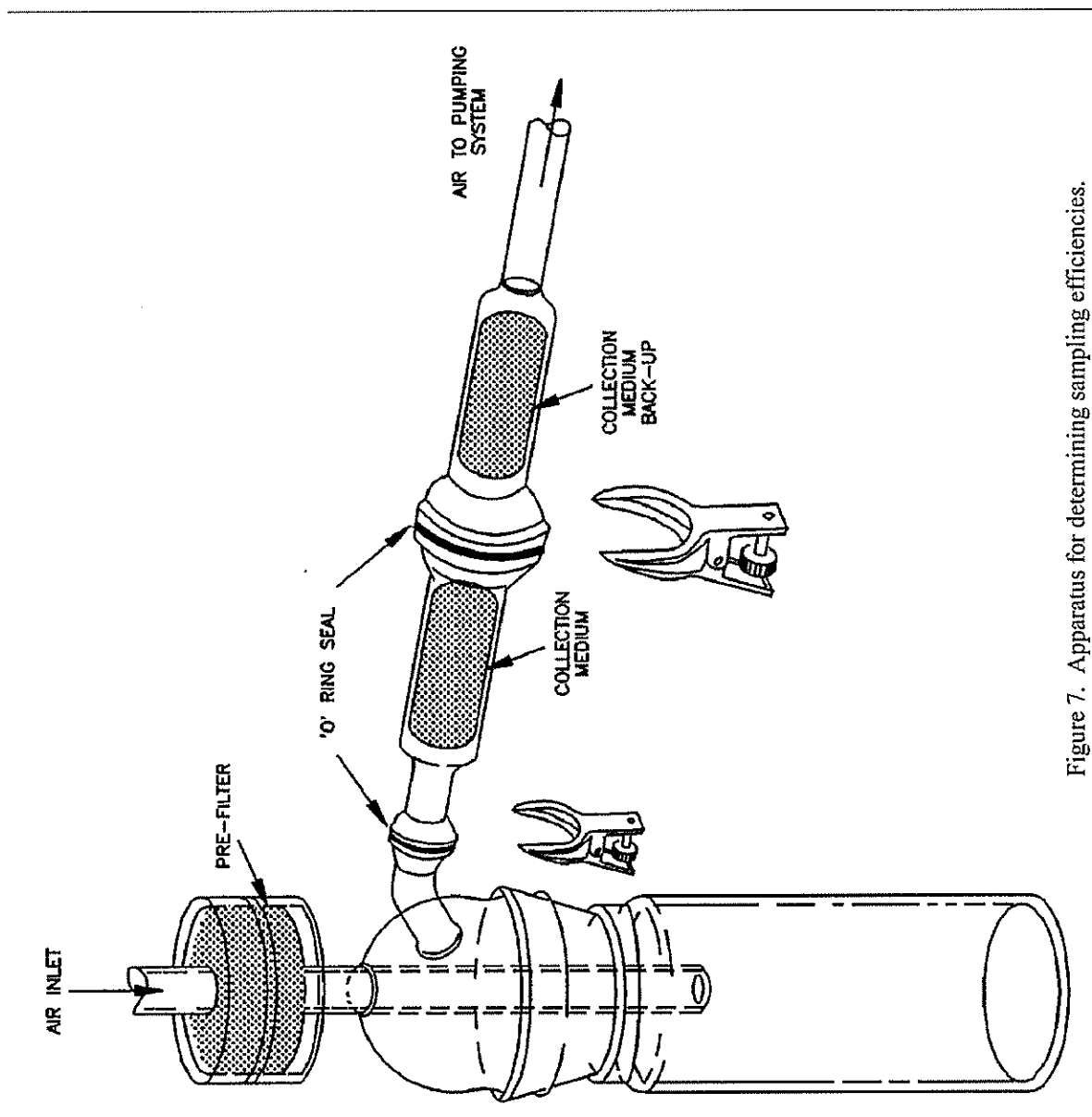


Figure 7. Apparatus for determining sampling efficiencies.

